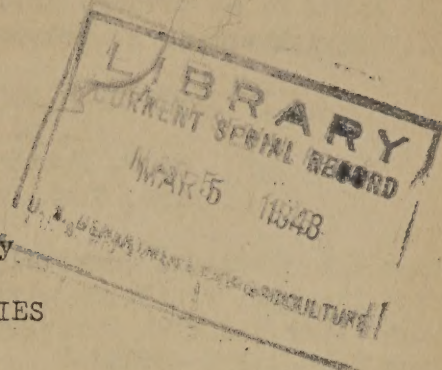


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U. S. DEPARTMENT OF AGRICULTURE
AGRICULTURAL RESEARCH ADMINISTRATION
BUREAU OF AGRICULTURAL AND INDUSTRIAL CHEMISTRY

Report on Research Activity
of the
REGIONAL RESEARCH LABORATORIES
in connection with
the Ninth Annual Meetings
of the



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COTTON UTILIZATION INVESTIGATIONS

COTTON LINT

Structure and Basic Properties of Fiber

The dimensional changes that take place in cross sections of cotton fibers upon mercerization and upon swelling with water have been studied in different varieties of cotton selected for fiber size and maturity. The effects of mercerization were observed in 16 varieties. Immature rather than mature cottons appeared to give the greater increase in cross-sectional area and the greater decrease in outside wall perimeter.

The validity of the differential dyeing test as a measure of wall thickness has been corroborated in several ways. One of the most striking examples occurred when a certain sample of Indian cotton produced fibers which dyed red at the base and green at the tip, indicating thick and thin cell walls, respectively. A single fiber of this type was therefore cross-sectioned at 350 points from base to tip. These cross sections bore out completely the evidence of the differential dyeing test.

Of the three indirect methods of measuring the swelling capacity of cotton by water retention, previously reported, the one in which the water is removed by centrifuging has given the most useful results to date. The method in which the water was removed by pressure has been definitely abandoned for a number of reasons -- one of the most serious experimental difficulties was the inability to prevent rewetting when the pressure was released. The method employing the rate of desiccation will be further investigated.

Measurements of the fiber strength of samples of cotton of known history have continued. The breaking strength of such cottons using different specimen lengths has been determined. This work has permitted estimation of the effective specimen length of the bundle used in the Pressley method. From data obtained on Stoneville and Wilds fibers this effective length lies between 1/16 and 3/32 inch.

The density of raw cotton has been measured by the density gradient method recently proposed for the study of plastics. The density of various raw cottons so determined varied from 1.535 to 1.543 gm./ml. When wax was removed by solvent extraction or when the cotton was mercerized, the density was lowered.

The discovery that grinding decreased the crystalline-amorphous ratio as determined by acid hydrolysis has been confirmed by measurements of accessibility (based on liberated glucose), heat of wetting, moisture absorption, and fluidity. No correlation with these results was apparent with X-ray measurements, but it is believed that the extent of the decrease was insufficient to be observed by this method.

Organic Acids in Raw Cotton

The relationship between the ash alkalinity of raw cotton fiber and the sum of pectic and other organic acids has been investigated. Close agreement has been observed in 7 out of 10 samples. In the other three, the ash alkalinity exceeded the sum of the acids by about 2 milliequivalents. The pH of the water extracts of these samples was 7.5 or higher. It is reasoned that the organic acids in these cottons had served as nutrients for microorganisms and were thus reduced, increasing the alkalinity of the water extract. This is being extended to include samples of cotton of known weathering or exposure before picking.

Freezing and Nonfreezing Water in Cotton

The investigation of the freezing and nonfreezing water in cotton fiber using the heat of fusion method is being extended to determine the influence of chemical treatments such as purification, mercerization, and acetylation. It has been established that pronounced differences observed in the proportion of bound water to total moistures between 5 and 30 percent depend upon the previous condition of the fiber sample, that is, upon whether equilibrium is reached by sorption or desorption; and that, for the given total water content, the percentage of nonfreezing water is greater when the moisture equilibrium is reached by desorption. Beyond a total moisture content of about 30 percent, however, the difference is negligible. These results have a bearing on the theory for the mechanism of the hysteresis phenomenon observed for sorption and desorption isotherms.

Fiber and Fabric Porosity

Pore-size distributions on fabrics of various constructions have been determined by dilatometric measurement. The curves show relatively smooth distributions between about 2,000 and 50,000 Angstrom units, with maxima varying between about 20,000-40,000. In this pore range the most probable locale of void spaces is within the yarn itself, namely, inter-fiber void spaces. This was borne out in a comparison of the distribution of two fabrics identical in thread count, weight per square yard, thickness, and yarn number, but with the significant difference that one was woven from yarn

spun from very fine, thin-walled fibers, while the other fabric was made up from yarn spun from more mature, thick-walled fibers. The pore-size distribution curve of the first fabric was in a lower range of pore radii than was that of the second fabric.

In two fabrics woven from the same yarn but differing from one another in that one contained 50 percent more picks per inch than the other, the pore-size distributions were nearly identical, in spite of the increased number of picks. This, too, indicates that the inter-fiber spaces within the yarn are the determining factor in the pore-size distribution between 2,000-50,000 Angstrom units.

Cotton Cellulose and Its Derivatives

The study of the properties of the cuprammonium complex with cellulose and with other carbohydrates has been continued. The findings in a wide variety of substances are being considered to discover whether any generalizations may be drawn concerning the structure in solution of sugar molecules and their derivatives.

A superior method has been developed for the determination of glycosidic methoxyl. This method appears to be specific for methoxyl in glycosidic linkages in small or large quantities in the presence of other methoxyls. It is being used in investigations of the structure and degradation of cellulose.

It has been shown that cotton yarns treated with liquid ammonia retain a greater proportion of their strength when heated than do untreated yarns. That this effect does not occur with the single fiber indicates the influence of changes in physical properties other than fiber strength.

Aminized Cotton

A method has been developed for the introduction of amino groups into cotton by use of 2-aminoethylsulfuric acid. Cotton fiber, yarn, or fabric is moistened with a 10 percent solution of this substance containing 25 percent sodium hydroxide. The treated material is heated for 40 minutes at 100° C. and then washed thoroughly. The nitrogen content of the cotton so treated is about 0.56 percent of the dry weight. The treatment only slightly reduces the breaking strength of the fabric. The treated fabric dyes readily with acid wool dyes. The amino groups serve as points for the addition of various elements or groups of elements to further modify cotton fabrics for particular end use purposes. This is illustrated by the addition of metallic elements to aminized cotton fabric to obtain rot resistance.

New Machinery for Opening Lint Cotton

The construction of a new type of mechanical opener designed to open up matted lumps or tufts of cotton to a loose and fluffy state is nearing completion. The new equipment will be connected with a line of cotton preparatory machines already in operation. The objective is the preparation of lint cotton for better cleaning prior to manufacturing than obtained with existing commercial machinery, without appreciable injury to the staple. If this machinery performs satisfactorily it should have application to the processing of mechanically harvested cottons.

Cotton Manufacturing Operations

A study has been completed of the effect on the strength and grade of a single coarse yarn (15.75 S) of changes in drafts during the drawing, roving, and spinning processes. Four different cottons varying in staple lengths from 1 to 1-1/2 inches were used. It was found that any appreciable increase in the spinning drafts definitely reduced strength with no effect on grade; but that neither the changes in drawing frame drafts and doublings nor in roving frame drafts caused any apparent loss in strength, or any change in yarn grade.

Rotproofing Cotton by Partial Acetylation

Research has continued on the rot-resistant cotton products prepared through the chemical modification of the cellulose by partial acetylation. In continued research on the chemical modification of cellulose by partial acetylation to produce rot-resistant cotton, emphasis has been upon the production of treated materials for trials in actual service.

Acetylated cotton cord, twine, and fish nets are undergoing tests in sea water and in the Great Lakes. Where reports have been received the modified cottons are proving much more durable than untreated cotton. Manufacture has been started on a small commercial scale of acetylated bags for use in domestic water-softening systems. An experimental fabric made up as a lining in shoes is being subjected to wearing tests. The reports of many of the tests are still to be received.

Since it appears advisable to ascertain more exactly the variations that occur in acetyl group distribution during the partial acetylation of cotton cellulose, as well as the influence that such variations may have upon the properties of the product, plans have been made for a detailed study of this phase of the subject.

Improving Weather Resistance of Cotton Fabrics

The information obtained in exposure tests of cotton fabrics during the past several years has led to a better understanding of the mechanism by which cotton is degraded under the influence of weather. This knowledge has been utilized in the formulation of more effective weather-resistant finishes, some of which appear to offer interesting possibilities for commercial development.

Experience has shown that cotton goods in the gray or loom state are more susceptible to rotting because of biological attack than are goods of similar construction from which the non-cellulosic constituents have been previously removed by processing steps such as scouring, kierboiling, or bleaching. Since under certain conditions of exposure in warm and moist climates biological damage to cotton textiles is often severe, it has generally proved advisable to employ commercially purified goods for out-of-doors use. This is less important with light goods of open structure such as tobacco cloth than it is with closely woven duck or canvas, since the former type of fabric loses its noncellulosic constituents rapidly from the action of rain, and thus offers less nutrition for micro-organisms.

Next to mildew, the photochemical action of sunlight appears to be the chief destructive factor in the weathering of cotton. The reaction here involved is believed to be oxidative in nature, the absorption of shortwave energy from sunlight markedly accelerating the normally very slow oxidation that occurs in cotton cellulose exposed to ordinary atmospheric conditions. Numerous mineral pigments are able to absorb solar energy, and thus may be employed as a protective finish for cotton textiles in exposure to sunlight. It has been found that a colorless urea-formaldehyde finish is also effective in protecting cotton materials against photochemical action; and that a finish containing a mineral pigment dispersed in urea-formaldehyde gives a higher degree of protection than does either component separately. For instance, a sample of untreated cotton duck suffered a loss of warp breaking strength of 40 percent, while a sample of the same fabric finished with a dispersion of lead chromate in urea-formaldehyde resin showed no strength loss at all. Samples of this fabric finished separately with lead chromate and with urea-formaldehyde each lost about 20 percent.

An examination of untreated tobacco shade cloth after a season's use in North Florida indicated that the damage suffered (about 50 percent strength loss) was almost entirely due to sunlight, with little or no evidence of biological attack. For this reason effort has been concentrated on the development of mineral pigment treatments of the ultraviolet-screening type which may be

applied to shade cloth on a commercial scale to meet the requirements of Florida tobacco growers for a more durable product. Since the type of cloth preferred by the Florida growers is too wide to be processed on regular machines available to the majority of cotton finishers, an attempt is now being made to treat the fabric in batches on standard laundry equipment. It is hoped that a considerable yardage of treated fabric may be made available during 1948 for field trials in Florida.

Improvement of Cotton Bleaching Methods

The work on bleaching has consisted very largely of a survey of current practices and has culminated in Department Technical Bulletin No. 941, entitled "Commercial Cotton-Bleaching Processes and Their Effect on Fabrics." The bulletin is intended particularly for reference to show the general level of present-day quality for comparison with the results from experimental bleaches or future commercial developments.

Cooperation with the American Association of Textile Chemists and Colorists on the Quartermaster Project (now discontinued) of obtaining the fastest possible OD-7 shade, was carried to the point of reporting the results of chemical and color analyses. This question of dyeing ties in so closely with the Southern Laboratory's research on swelling and self-sealing fabrics that the research will be continued here.

A long-range project on the effects of storage on bleached goods is continuing.

Use of Cotton for Tire Cord

The development of improved types of cotton tire cord has continued to be a major line of research at the Southern Laboratory. The liaison customarily maintained with industry has recently been improved by appointment of collaborators from the major tire manufacturing companies. Two profitable conferences were held at the Laboratory during 1947 with subsequent meetings planned at 6-month intervals.

New stretching device. When cotton tire cord is removed from the final twisting machine, it has a higher elongation at rupture and at various loads than is desirable. With a view to providing a better machine for removing this excessive elongation, a stretching device has been perfected which subjects the cord to a constant tension during stretching, instead of to the constant percentage of stretch applied in present commercial practice. With the new machine, segments of the cord having a high original elongation are pulled down more than segments having a lower elongation, and there

is a tendency to cancel out any nonuniformity of elongation present in the unstretched cord. With the commercial methods, obviously a cord having a higher original elongation than another cord will have less elongation taken out when both are subjected to the same percentage of stretch, with the tendency to pass on into the finished cord any nonuniformity in elongation present in the unstretched cord.

A number of tire and tire cord manufacturers are extremely interested in the new stretching device and several have sent representatives to obtain first-hand information. Cooperative experiments are now being carried on to obtain full information regarding its commercial possibilities.

Service tests on large-size tires. Claims have been repeatedly made that satisfactory, large-size, synthetic rubber tires for highway service cannot be made with cotton cord. Partly to check such claims, but particularly to obtain performance data as a guide for research to develop improved types of cotton cord, the Southern Laboratory secured the cooperation of a large tire manufacturer in building four groups of 9.00-20, S-6 (about 70 percent synthetic rubber) experimental tires for highway service tests. One group was made with regular-production cotton cord, two groups with experimental cord made from Wilds cotton by different processes, and one group from regular-production rayon cord (this last group for purposes of comparison). Through the cooperation of four New Orleans trucking firms, a tire from each group was placed in four different classes of service. Accurate records were kept of the loads carried on each trip, and measurements were taken at regular intervals on treadwear, growth, operating temperatures, and air-pressure build-up.

One group of the tires, mounted on the trailer of a truck, was run at moderate speed underloaded, and was still in operation at 148,000 miles. A second group, mounted on the rear tractor wheels of a truck, was run at normal loads and moderate speeds, and was still in service at 55,000 miles with no failures and one recapping. The third group, tested on the rear tractor wheels at overloads at high speeds, is out of service, one tire having been removed at about 18,000 miles owing to an injury and the others having successfully taken two recaps for a total of 60,000 miles -- more mileage than the trucking company concerned ordinarily runs tires in this class of service. Of the fourth group of tires, operated on the rear tractor wheels overloaded at high-speed, two cotton-cord tires were removed at 18,000 miles owing to injuries; another cotton-cord tire took two recaps for a total of 59,000 miles before failure of the carcass and the one rayon tire remains in operation after 79,390 miles.

It is of interest that these tests, which have provided the useful performance data needed, also have indicated that even standard commercial cotton cords are entirely satisfactory for use in large tires in high-speed, moderately overloaded service.

Economic and Technological Surveys and Appraisals

Southern Laboratory personnel contributed to the research program report entitled "Study of Agricultural and Economic Problems of the Cotton Belt," published by the Cotton Subcommittee of the House Committee on Agriculture in 1947. They participated in summing up the facts and conclusions in the projects of this Subcommittee relative to the marketing of cotton, cotton's competitive position in end-use markets, and the impact on cotton of competition from synthetic fibers and paper.

In connection with the latter study, a survey of the opinion of textile chemists and engineers indicated that cotton at present is decidedly superior to rayon in launderability and wet strength, and is superior to it in such properties as fastness to dyes, resistance to abrasion and dimensional stability; but that, if present research trends continue, cotton will lose at least part of these advantages in the next few years. In the summary of the study it was stated that cotton has lost important end-use markets during the last 15 years to rayon and paper, and faces the prospect of greatly intensified competition from them in the future, as well as from the synthetic fibers other than rayon, only now beginning to be produced in quantity. It was concluded that if consumption of cotton is to be maintained at anywhere near present levels, cotton must have a competitive price and be supported by much more aggressive research and merchandising programs than in the past.

Comprehensive basic information on the consumption in the United States of cotton, wool and related fibers, silk, flax, rayon, other synthetic fibers, jute, the various hard fibers and hemp, has been compiled for the period 1892-1946. The data reported indicate that, following a level or only slightly rising trend between 1918 and 1940, there was a sharply upward trend in the consumption of fibers in the United States during World War II. Although fiber consumption has declined since the peak period of 1941-42, during 1946 it was greater than for any other peacetime year. Of the total quantities of fiber made available for ultimate consumption in 1946, cotton comprised 61 percent; rayon, 11 percent; wool, 10 percent; jute, 10 percent; hard fibers, 6 percent; and silk, flax, other synthetic fibers and hemp, less than 1 percent each. Per capita consumption of fibers failed to show any sustained increase during the long period from 1905 to 1939, but jumped to record heights during World War II, and in 1946 remained far higher than during any

prewar year. A preliminary survey for 1947 in connection with the study, indicated that total mill consumption of all fibers was as great in that year as in 1946, but that because of unprecedented exports of textiles, the quantity remaining in this country for ultimate consumers was sharply down from the previous year, particularly so for cotton.

COTTONSEED

Improving Storage Properties

Investigations of the chemical method of controlling deterioration of cottonseed have been continued. In laboratory-scale tests 30 organic chemicals have been found which are biologically active when applied to cottonseed in small amounts. These will be used in further research on the nature and behavior of the enzyme systems of cottonseed and other oil-bearing materials. Their effect on bacteria in relation to spoilage will also be investigated. A determination of effect on viability is planned in cooperation with the Mississippi Agricultural Experiment Station.

Three mill-scale experiments using 1946 cottonseed and flaxseed (South Texas) crops were completed in which the effectiveness of ethylene chlorhydrin alone or mixed with chloramine-T in inhibiting heating was demonstrated. The treatment was more effective in seeds having a moisture content of 22 percent or higher, and less so in seeds of lower moisture content.

Two mill-scale experiments using 1947 cottonseed crop have been completed and another is still under way. In the completed tests the treatment consisted in spraying the seed in a closed conveyor as it moved to the bin with a mixture of propylene glycol dipropionate and dimethyl bischloromethyl benzene, in a ratio of 15:1 at the rate of 12.5 pounds of chemical mixture per ton of seed. The two experiments demonstrated that this particular mixture effectively inhibited heating and the formation of free fatty acid in cottonseed having a moisture content of about 16 percent. There was a high correlation between the results obtained in the mill-scale tests and those obtained on seeds from the same lot tested in the laboratory by the rapid-screening technique.

Experimental work on the storage of cottonseed under conditions which minimize changes has been completed. The results show that dry cottonseed (less than 8 percent moisture) may be stored at -18°C . and perhaps at 1°C . without appreciable change in chemical composition. Prior to this work two indexes of deterioration were available, that is, germination and free fatty acid content of the oil. The present work demonstrates the usefulness of increase in the "red gossypol" of Podolskaja or the

gossypurpurin of Boatner and the increase in nongossypol absorption at 2360 Å as indexes of deterioration. The increase in absorption may be due to a change in the diene-conjugation of the oil. Since peanuts also show this change in storage, it may be true of oilseeds in general during storage.

Properties of Pigments

New data continue to be obtained on the behavior and occurrence of the pigments of cottonseed. The influence of species and variety on the content of gossypol and gossypurpurin in cottonseed grown under identical conditions (by the Bureau of Plant Industry, Soils, and Agricultural Engineering) was investigated. Samples were analyzed for contents of moisture, nitrogen, gossypol and gossypurpurin, after which they were stored under identical conditions and analyzed for gossypol and gossypurpurin at fixed intervals over a period of a year. There was no apparent correlation between pigment content and length of fiber or lipid and nitrogen contents. However, a definite relation was found between species and gossypol and gossypurpurin contents, even though there was a wide variation in the pigment content of different varieties of seed in the same species or subspecies. There was considerable variation but no consistent pattern of change in the gossypol content. On the other hand the variation in gossypurpurin content followed a consistent pattern: the pigment increased rapidly the first 2 to 3 months of storage, and less rapidly thereafter. Since the amount of pigment is probably influenced by environmental factors, a selection of varieties under different conditions of growth will be required for further investigation.

Color analyses of oils obtained in commercial operations under different conditions of processing showed that hydraulic-pressed oils from wet-cooked seed had a lower initial bleach color and a less rapid color increase during storage of the oils than had screw-pressed oils for dry-cooked seed. To understand better this behavior of seed during processing a series of laboratory-scale cooking experiments was carried out. From these it was concluded that the conversion of native cottonseed pigments to more highly polar compounds during cooking of the seed in the presence of moisture is the result of reactions which occur within the pigment glands rather than in the extra-glandular tissues, as has previously been postulated. The oils from seed cooked with different amounts of added water and with varied time and temperature, were examined in respect to refining and bleaching performance. Oils of least bleach color were obtained from seed cooked in the presence of relatively large amounts of added water (10-15 percent) and at relatively high temperatures (235°-244°F). The feasibility of adopting this set of conditions in commercial operations, however, may depend upon the effect on the nutritional value of the

meal obtained..

Solvent Extraction

Research on solvent extraction of cottonseed has proceeded along a broad front -- laboratory studies to improve processing methods, pilot-plant runs to test equipment and operating conditions, and design of improved units for process development research.

An important step -- the treatment of flaked meats to minimize formation of fines -- has been investigated on a laboratory scale by a percolation technique devised for the purpose. While some data have been obtained on the best combination of heat and moisture, the problem may still be far from final solution for seed of different histories. The correlation of results obtained by this laboratory percolation technique with those obtained in pilot-plant operations has not yet been established.

Four pilot-plant-scale runs were made to test various units involved in solvent extraction operations and to determine optimum operating conditions. The oil and solvent recovery plant performed in a satisfactory manner but difficulties encountered in the operation of the extractor and meal dryer have necessitated the redesign and alteration of parts of these units. These alterations are underway. Over 11,000 pounds of flaked cottonseed meats were used in this research.

Over 31,000 pounds of cottonseed have been cleaned, delinted, hulled, and purified in the cottonseed and peanut preparation plant to obtain the quantity of meats needed in process development research as well as for the production of oil, glands, and meals required in other research investigations.

COTTONSEED OIL

Spectrochemical Analysis of Vegetable Oils

A spectrochemical method for the determination of trace elements in vegetable oils has been developed which permits determination of as little as 1 part of the trace metallic elements in 10 million parts of oil. The method has been applied to some 40 types of vegetable oils and fats for the determination of copper, iron, manganese, nickel, and tin. Traces of each of these metals have been found in at least some of the oils, the concentration varying from zero parts per million to exceptional cases of as much as 30 parts per million. Hydrogenated oils frequently show presence of hydrogenation catalysts

in the order of magnitude of 5 parts per million, and some refined and bleached oils reveal the presence of metallic contamination from the equipment used in the process or from the containers in which they were stored.

SWEETPOTATO UTILIZATION INVESTIGATIONS

Commercial-Scale Sweetpotato Starch Processing

The sweetpotato starch manufacturing plant of the U. S. Sugar Corporation was operated from December 1946 to March 1947; but production was at reduced capacity chiefly because of insufficient raw material. Additional technical studies of starch production during this period of operation further demonstrated that the basic flow sheet developed by the Bureau was sound, and that the design, equipment, and performance of the plant were in general good.

At the close of this period, corporation officials announced suspension of further starch processing operations until solution of the problems encountered in field production of the crop. These problems, entirely agricultural, are essentially of the same nature as those encountered in the starch enterprise at Laurel, Mississippi, and as those which impede further development of sweetpotato starch enterprises elsewhere. In the Mississippi undertaking, the cost of growing sweetpotatoes exceeded the price that the factory could afford to pay for raw material without the aid of Government subsidies. In the Florida undertaking it was estimated that the long growing and harvesting seasons and other favorable conditions would assure sufficiently heavy sustained yields to reduce crop production costs to less than \$10 per ton, even with relatively high costs per acre. However, when production was scaled up to the volume required to support the factory, difficulties not forecast by the results of extensive experimental planting were encountered. Yields were much lower than estimated and costs were excessive.

Byproduct Recovery and Utilization

Research has been continued on the recovery and utilization of natural protein and production of feed yeast from sweetpotato starch processing waste waters.

Refinements have been made in the pilot-plant processes and equipment for coagulating, settling, and dewatering the natural protein concentrate. In large-scale experimental runs, sufficient crude protein and byproduct pulp were obtained to produce 800 pounds of an enriched pulp containing 11.5 percent crude protein and 800

pounds of unfortified pulp containing 1.8 percent crude protein. During November 1947 the Department of Animal Industry of the Florida Agricultural Experiment Station began feeding trials on calves for the preliminary nutritional evaluation of these materials.

The pilot plant designed and built at the Southern Laboratory for the production of feed yeast from the sugars in sweetpotato fruit water has been further tested on citrus press liquor in production runs carried out by the Agricultural Chemical Research division in cooperation with the Dr. P. Phillips Canning Co. of Orlando, Florida. Details are given in another section of this report.

Removal of Sugars and Proteins by Diffusion Processes

Renewed pilot-plant investigations were undertaken to obtain added data on the application of diffusion processes to facilitate separation of solubles and recovery of byproducts in conjunction with simplified sweetpotato starch extraction and purification processes.

A series of diffusion experiments were also carried out in the laboratory on 2-kg. samples and in the pilot plant on 200-pound samples of cossetted L-5 sweetpotatoes, both stored and fresh-dug, and with temperature and alkalinity of water systematically varied. Whereas over 75 percent of the protein and practically all of the sugars are dissolved and appear in the first fruit water when sweetpotatoes are ground with water directly and screened in conventional equipment, only 40 percent of the protein and 60 percent of the sugars were removed by diffusion of cossetted potatoes. When the cossettes were pretreated with gaseous carbon tetrachloride as a plasmolyzing agent, the extraction of sugars was almost complete but only 47 percent of the protein was removed.

Because the separation of nitrogenous constituents is inadequate and the recovery of high-quality starch more difficult, the aqueous diffusion of cossetted sweetpotatoes does not appear to meet the requirements of a simplified process for removal of soluble nonstarch constituents in sweetpotato starch processing operations.

Comparative Qualities of Different Varieties of Sweetpotatoes

Samples of 25 varieties of high-starch sweetpotatoes and of the same number of table varieties, from the 1946 variety test plots of the Mississippi Agricultural Experiment Station, were analyzed for starch. It appeared that in high-starch sweetpotatoes grown in the same location, variation in the percentage of moisture was the major factor causing variation in percentage

of starch. When reduced to the same moisture basis, the starch contents of three leading varieties of high-starch sweetpotatoes have been nearly identical for the past several years.

The purified native starches from 22 domestic varieties of sweetpotatoes have been examined for amylose content. The group included established and new varieties of the starch and table types which exhibited a considerable range of variation in superficial characteristics. The apparent amylose content of the non-defatted starches ranged from 17.5 to 21.7 percent with an average of 20.3 percent. In 16 of the 22 varieties the value fell in the range of 20.0 to 21.5 percent. Although the results do not encourage the prospect of discovering a trend toward high amylose content in any existing variety of sweetpotato the quest should not be dropped in view of the higher industrial value of starch composed chiefly of straight chain amylose.

PEANUT AND OTHER OILSEED UTILIZATION INVESTIGATIONS

PEANUTS

Solvent Extraction

About 10,775 pounds of flaked peanut meats have been processed with commercial hexane in continuous solvent-extraction equipment and auxiliary units for oil and solvent recovery. None of the runs has been successful in producing an extracted meal of suitably low oil and solvent content; but a product of satisfactory lipids content was obtained by reextraction of meats. A good deal of useful information was obtained on the nature of the operational problems in the solvent extraction of peanuts.

Research has continued on the preparation of flakes of proper toughness and stability for efficient handling during solvent extraction. A laboratory procedure was developed to determine in advance the practicability of a flake for pilot-plant extraction using porosity as the criterion, with porosity judged by the time taken for fixed increments of solvent to percolate through a set volume of flakes. This method was used to compare flakes prepared from (1) moistened meats which had been heated before flaking but with no additional heating, and (2) from meats moistened to the same level as those of the previous series and heated before flaking plus heating in hot air after flaking. The heating with hot air made the flakes objectionably fragile, but gave faster percolation rates and flakes lighter in bulk density. So far the combination of moisture and heat treatment before flaking appears to give the best conditioning of the material for experimental solvent extraction. Many more types of flakes will have to be tested before behavior in pilot-plant operations can be accurately predicted.

PEANUT OIL

Improvement of Stability

Norconidendrin, a chemical prepared from the sulfite waste liquors of western hemlock, was further investigated as an antioxidant for hydrogenated and unhydrogenated oils and fats. While in general norconidendrin was slightly less active in hydrogenated than in unhydrogenated oils, addition of only a small quantity of norconidendrin even to the hydrogenated oils significantly improved keeping quality.

For comparative purposes norconidendrin was tested in lard and showed good activity. Tests of norconidendrin in the presence of other antioxidants showed that the addition of a-tocopherol reduced the effectiveness of norconidendrin. It was found that norconidendrin was neither removed nor destroyed during the deodorization process.

Studies of the structure of this compound indicated that the basic structure of the parent substance, conidendrin, survived demethylation; but with, apparently, an alteration in the functional side chain. Experiments on the recovery of conidendrin from the waste liquors indicated that recovery was feasible by low-temperature vacuum methods, followed by extraction with ethylene chloride, and may be a possibility for commercial economy in the production of conidendrin.

Samples of norconidendrin have been prepared and sent to numerous industrial firms for further testing and evaluation.

PEANUT MEAL AND PROTEIN

Identification and Removal of Nonprotein Materials in Protein Preparations.

The nonprotein material in peanut protein prepared by the usual methods from solvent-extracted meal has been found to consist of sugars, nonprotein nitrogenous substances, lipids, and ash. The first three groups of substances can be removed to a large extent by washing the protein with alcohol in the wet curd state; more efficiently when the curds have not been coalesced by dewatering.

Protein Production

With the objective of aiding industrial concerns which are getting ready to use peanut protein in manufactured articles, peanut protein has been produced in the pilot plant of the Southern Laboratory. Experiments were conducted using 100-pound batches of

solvent-extracted peanut meal to determine the optimum conditions for carrying out the special process used to extract the protein. In the series of runs average yield was 40 percent protein, typically containing 16.3 percent nitrogen, 1.2 percent ash, 0.5 percent phosphorus, and 0.8 percent sulfur.

Fiber Production and Processing

Previous reports from the Southern Laboratory have described the production of a fiber from peanut protein. This fiber proved practicable enough to permit further development by industry of the production process. Recently the Virginia-Carolina Chemical Company has reported semi-industrial manufacture of the peanut protein fiber "Vicara." From the beginning this company has followed the research developments at the Southern Laboratory. At least two other firms are preparing to produce the fiber. Over 527 pounds of peanut materials prepared at the Southern Laboratory have been shipped to a cooperating firm for fiber investigations. Fundamental research on peanut protein fiber to improve it still further for industrial uses is being continued.

Paper Coating from Peanut Protein

The paper industry offers a good outlet for peanut protein as a binder in coatings. It has been found at the Southern Laboratory that a satisfactory paper coating can be made with peanut protein as an adhesive for a wide variety of pigments. Use of neutralized protein dispersions gives a light-colored coating having a wax-pick value equal to that obtained with other commercial proteins.

Factors determining the quality of a protein paper coating have been investigated in three different types of peanut protein preparations. From tests of more than 180 peanut protein coating formulas on over 2000 sheets of raw paper stock, it appeared that the wax-pick test was directly related to number of parts of protein used per 100 parts of pigment; that decreasing the percentage of the total solids of the slips and holding the pH constant, increased wax-pick; and that decreasing the percentage of the total solids but decreasing the pH of the slip maintained the wax pick constant. Coated papers have been prepared with a wax-pick test range of 3 to 13, the pH varying from 6 to 12.5; the percentage of solids of the slip varying from 15 to 45 percent; and the concentration of protein varying from 5 to 40 parts per 100 parts of pigment.

RESEARCH AND MARKETING ACT INVESTIGATIONS

Research has started at the Southern Laboratory on the five work projects approved under RMA authority. The progress made along the lines planned under each project is reported, with objectives briefly indicated.

RMA PROJECT 21 Develop New and Improved Uses for Rice, Rice Bran, and Better Methods of Processing and Handling Prior and Subsequent to Processing

(1) Investigations of the effect of processing factors on the composition and industrial utility of rice bran oil produced from southwestern rice. When freshly milled rice bran of good quality is extracted with commercial hexane an oil of relatively low free fatty acid content having a good color and as stable as other similar types of crude oils, is obtained. Some of the rice bran oils when treated in the laboratory according to usual practice yielded products acceptable for use in the edible trade.

RMA PROJECT 102 Fundamental Characteristics of Cotton Fiber as a Means of Developing Entirely New Uses

(1) Microscopical evaluation of the swelling of fibers as a basis for selecting cottons for new types of protective fabrics. The swelling of individual cotton fibers is being investigated by the method developed in this laboratory of comparing cross-sectional areas wet and dry. The relative fiber swellabilities of certain representative varieties of cotton will be determined by averaging several hundred individual measurements for each type. These studies are expected to aid in selecting cottons for new types of water-resistant fabrics.

(2) Study of changes in the fiber upon oxidation. A task force will have to be assembled to conduct this investigation. A few members of the force are ready to enter upon work and others engaged are awaiting routine approval. The information obtained will guide the formulation of treatments to prevent deterioration of cotton by oxidation.

(3) Chemical bonding of cotton fiber with resins to produce new cotton materials. Arrangements have been completed for the loan of a Single End Fiber Bonding Laboratory Machine for use in the initial investigations.

(4) Improvement of elastic recovery of cotton fiber by chemical modification. A survey of the literature made and preliminary experiments have been carried out on the effect of a few chemicals on the physical and chemical properties of the fiber.

(5) Energy relations in performance of mechanical cotton goods. Preliminary work has dealt with the evaluation of a sharp flex test procedure in relation to energy performance in mechanical goods. Under construction is a new machine of this type able to test four samples at a time.

(6) Estimation of immaturity in cotton by means of dyeing. The differential dye test developed by the Southern Laboratory for distinguishing between immature (thin-walled) and mature (thick-walled) is being investigated from the fundamental viewpoint for the purpose of perfecting the test. In cooperative work with the Cotton Branch of Production & Marketing Administration, it is hoped to develop color standards to represent degrees of maturity.

RMA PROJECT 103 Develop New and Improved Products from Cottonseed and Other Cotton Products

(1) Composition of Cottonseed as influenced by environment and variety. Samples of cottonseed from 8 varieties grown at 13 stations in the cotton producing areas have been received and catalogued. Additional samples from four of the stations will extend the list of varieties. Analysis of the samples is to be started in early 1948.

(2) Pilot-plant fractionation of cottonseed kernels into oil, meal, and pigment glands. Pre-pilot plant work has been carried out on the 3-way fractionation of kernels to obtain information on behavior with this principle of processing and to produce materials for use in other research. The potential usefulness of the new products justified investigation of the unit operations on a pilot-plant scale with the idea of developing a commercially feasible process. Engineering studies have been started on disintegration of flaked kernels in solvent slurries using equipment which detaches the pigment glands from the meal by fluid friction. Cooperative agreements have been completed with several commercial firms to test their equipment in various unit operations.

(3) Utilization of pigment glands and derived products. In a cooperative investigation at Columbia University, rats given gossypol by stomach tube lost weight rapidly and finally died of starvation. Since no symptoms other than those of starvation were found death was attributed to the loss of appetite resulting from the delayed emptying of the stomach caused by gossypol. Parallel pharmaceutical investigations are underway on intact pigment glands and on isolated constituents. At the Southern Laboratory pigment glands are being fractionated and constituents characterized with a view to determining chemical and pharmaceutical applications.

(4) Investigation of the nutritional value of cottonseed byproducts produced by new methods of fractionation. Preliminary experiments by the Bureau of Animal Industry have shown the excellence of deglanded cottonseed flour as the sole source of protein in feed for chicks and laying hens. Some retardation of growth occurred which was presumably due to the physical condition of the flour. While further nutritional studies are being conducted by various cooperating laboratories, the Southern Laboratory has undertaken chemical examination of this flour to detect constituents which might affect nutritional value.

(5) Pilot-plant processing of cottonseed oils and their derived products. The installation of equipment for refining, bleaching, deodorizing, fat-splitting, and related operations on cottonseed and other vegetable oils is nearing completion.

RMA PROJECT 104 Development of New and Improved Products from Cotton Fiber Through Processing and Chemical Treatment

Personnel and equipment are being acquired to carry out mechanical processing research in two lines of work:

(1) The effect of twist on the efficiency of weaving and on the quality of woven fabrics.

(2) The development of cotton fabrics and garments of good warmth qualities.

In the field of chemical processing the three following investigations have been started:

(1) The development of improved plastic laminates with cotton as the filler. A task force has been organized for the purpose of determining as accurately as possible the effect of yarn and fabric construction upon finished laminates.

(2) Improvement in the resistance of cotton bagging fabrics to penetration by insects and rodents. A test by the Bureau of Entomology and Plant Quarantine on cotton products treated by the Southern Laboratory showed that insects did not penetrate bagging containing as little as 0.25 percent DDT. Current investigations are directed toward improving the application of DDT and the discovery of other effective repellents.

(3) Development of swelling-type water-resistant fabrics. Exploratory experiments have indicated that thin-walled fibers are more effective than thick-walled fibers for this type of fabric. Current investigations are directed toward determining the significance of differences in fiber size, shape, wall thickness, and swelling capacity.

RMA PROJECT 124 Quality Improvement of Peanut Products and New Uses for Peanut Oil

(1) Development of improved peanut butter. On order is peanut butter manufacturing equipment with which to conduct research on factors affecting the quality of peanut butter.

(2) New uses for peanut oil. Pilot-plant facilities installed for oil processing research under RMA will be used on peanut oil as well as on cottonseed, and rice bran oils. It is planned to develop low-temperature crystallization techniques for fractionation of these oils.

WESTERN REGIONAL RESEARCH LABORATORY
M. J. Copley, Director

ALFALFA UTILIZATION INVESTIGATIONS

DEHYDRATION

Alfalfa Dehydration, History of the Industry

To determine the economic position of dehydrated alfalfa meal, a study is under way covering the geographical location of the industry, the development of grades and standards for the product, and a statistical record of prices and production. Production has grown to an annual output of over a half million tons with increasingly better prices for high quality dehydrated meal. Over 200 commercial plants have been identified. Their locations indicate a tendency toward concentration in the middle Mississippi Valley, particularly the portion in southern Missouri and eastern Arkansas; the Great Plains area, embracing central and eastern portions of Nebraska, Kansas, and the central portion of Oklahoma; the Central Valley of California; the Imperial Valley of California; the Roswell (New Mexico) region; and southeastern Colorado.

Production Economics of Dehydrated Alfalfa

From data available in the literature and from equipment manufacturers, a cost estimate for producing dehydrated alfalfa meal was made for a typical plant. It was assumed that the plant would operate at a rate of one ton of dry meal per hour and produce 2400 tons of meal per year with an operating season of about five months. The plant investment was estimated to be about \$60,000, and the production cost (exclusive of raw material but including harvesting from the field and bagging) was estimated to be approximately \$26.50 per ton of meal.

Since dehydrated alfalfa meal is finding increasing acceptance and high-grade meal commands a price differential which takes account of its quality, increased emphasis on studies directed toward quality improvement appears to be justified.

The Isomers of Carotene

In the development of a rapid, accurate method for routine analysis of carotene in alfalfa extracts, it has been necessary to prepare pure crystalline stereoisomers for standards. All-trans-beta-carotene, all-trans-alpha-carotene, neo-beta-carotene U, and neo-beta-carotene B were isolated and purified by chromatographic and crystallization techniques. The absorption spectrum of each was determined in iso-octane and commercial hexane in the range of 320 to 500 millimicrons.

The specific absorption values obtained are considered suitable as a basis of analysis for individual isomers. This step is important since biological activity and nutritional values of the

isomers are reported to differ markedly. The use of the technique developed for the separation of isomers and the application of the constants determined for these isomers makes possible more accurate evaluation of deteriorative changes in alfalfa.

Fractions from Alfalfa for Poultry Feed Tests

Preliminary results have been obtained on a fresh-alfalfa fraction which was used in poultry feeding experiments in the Poultry Division of the University of California at Berkeley. The fraction tested was prepared by disintegrating alfalfa, expressing the juice, removing the heat-coagulated protein by filtration, and evaporating the juice to dryness in a vacuum. This fraction, containing 21 percent protein (N x 6.25) and no fiber, was fed without retarding growth at a feed level equivalent to a ration containing 50 percent of alfalfa meal. In general, alfalfa meal retards chicken growth when fed at levels greater than 10 percent of the diet by weight. These results suggest a method for increased use of alfalfa in poultry rations.

Lipids and Phospholipids

By saponifying the acetone-soluble portion of alfalfa lipids and determining the glycerol content of the saponificate, it has been possible to arrive at an estimate of the probable distribution of the fatty acids present among the three classes of compounds: triglycerides, phospholipids, and wax esters.

Subsequently, an attempt was made to obtain a similar fatty-acid distribution for an acetone-insoluble fraction of the alfalfa lipids; but in this case, carbohydrates were found present in so great a degree that the glycerol analysis was completely out of line and it was impossible to work out even an approximation of the relative amounts of fatty derivatives. Means were considered for separating the glycerol by distillation prior to analysis but this step would require an elaborate procedure involving introduction of new sources of error.

During the course of the investigation on alfalfa lipids, various measures were tried for eliminating the large amounts of pigments from the different fractions. No success was obtained with any of a number of representative selective absorbents, and attention was subsequently turned to a study of solvents in the hope of finding one, or possibly a combination, that would yield a reasonably sharp separation, particularly of the chlorophyll. It was found that if crude alfalfa lipid extracts were treated with acetonitrile and the resulting solution in turn extracted with petroleum ether, a high content of carotene, and practically no chlorophyll, was obtained in the latter solvent. The solution was a clear yellowish orange and had a carotene content of 5.0 percent based on the weight of total solids.

It was found possible to achieve quantitative extraction of carotene from fresh alfalfa by maceration with cold acetonitrile.

Moreover, the carotene would be quantitatively transferred from the nitrile to petroleum ether by simply washing the former with the latter. Finally, the relatively small amount of green pigment dissolved in the petroleum ether could be removed by washing it with several portions of fresh nitrile. Similar treatments applied to dehydrated alfalfa led to similar results.

FRUIT UTILIZATION INVESTIGATIONS

FREEZING PRESERVATION

Prefreezing Treatment of Apricots, Peaches, Apples

With apricots and peaches, excellent color was obtained with either of the following prefreezing dipping baths: 2 percent sodium chloride plus 1 percent ascorbic acid or 1 percent sodium chloride, 0.5 percent ascorbic acid, and 500-1000 ppm. of sulfur dioxide (added as sodium bisulfite). The pH of the latter solution was adjusted to 3 with citric acid. Analyses of baked fruit revealed 10-20 mg. of ascorbic acid per 100 grams of fruit and a sulfur dioxide concentration of less than 20 ppm. The small amount of salt appears to have a synergistic effect when used with ascorbic acid, and the small amount of sulfur dioxide increases the effect. These findings may be of considerable practical importance to commercial producers.

In studies with apples, slices of the Delicious variety were filled with solutions of various calcium salts, including 0.1 to 1.0 percent of calcium chloride. The slices were filled by immersion in the solutions, application of 29 inches of vacuum, and release of the vacuum. On being heated, as in baking in pies, they exhibited increased firming, but an apparent objection was their considerable absorption of water.

Filling the slices by vacuum with a concentrated (60 percent) sucrose solution, however, produced a crisp, firm texture, free from objectionable flavors and excess water. Reducing the sucrose to 50 percent or 40 percent did not affect the texture significantly. Dipping in a solution of sodium bisulfite (1000-2000 ppm. SO_2) adjusted to pH 3 with citric acid for 10 seconds prior to the sirup-vacuum procedure prevented discoloration for 8 hours after defrosting.

About 15 percent of the Delicious apple crop is grade C and would be suitable in large part for use in pies. The processing technique including absorption of sugar sirup, possibly with added citric acid and ascorbic acid may prove advantageous.

Susceptibility of Peach Varieties to Browning

A total of 51 varieties of peaches and nectarines obtained from the California Agricultural Experiment Station at Winters, California, have been analyzed for their browning characteristics.

The more promising varieties were packed in 40 percent sucrose sirup and subsequently appraised for color, flavor, and texture. The results obtained were essentially the same as those reported for the 1946 season. The extent of browning which occurred in most varieties was directly correlated with the total tannin content.

The total tannin, expressed as milligram percent tannic acid, was determined on an unoxidized frozen sample of each variety. Corresponding frozen peach samples of each variety were peeled, sliced, and exposed to the air in flat pans at approximately 20° C. and observed at intervals over a period of 24 hours. None of the slices of the four varieties with lowest tannin content in the range of 32 to 38 milligram percent showed any browning at the end of 24 hours. Three of these four varieties were of the same cross; Sunbeam x Rio Oso Gem. Samples of the six varieties with tannin content ranging from 40 to 54 milligram percent were very slightly brown at the end of 24 hours, with traces of discoloration appearing within 4 to 5 hours. These results suggest that a concentration of 39 milligram percent is critical with respect to the appearance of brown discoloration. Peaches containing less than this amount showed no significant browning while peaches containing more were susceptible to browning with the developed color intensity roughly proportional to the original tannin content. Analyses of more varieties will be made to determine the validity of the stated critical value.

A number of varieties of peaches, some similar to those listed above, have been harvested and frozen at the Irrigation Branch Experiment Station, Prosser, Washington. The samples have been brought to the Western Regional Research Laboratory for these investigations.

Bacteriology of Orange Juice

The coliform index in orange juice from nine orange juice plants in Southern California showed great variation in samples from different plants, from 0 to 1100 per 100 cc. of juice. The highest count was found in a plant considered to have very high sanitation standards. An observation was made that 12 of 14 samples of juice that had an excess of peel oil, owing to faulty reaming, contained no coliforms. This suggests the possibility that the peel oil contains some germicidal principle. In tracing the source of coliform infection, it is important to differentiate the "fecal" from the "non-fecal" coliforms. Since no satisfactory rapid method of differentiation exists for coliforms in orange juice, experiments were carried out to test the applicability of two rapid methods, the Koser's citrate and the Eijkman's tests that are currently used for shellfish and shellfish waters. The results were compared with the standard IMViC tests and have shown that in 90 coliform cultures the incidence of false differentiations was 6 percent with the Koser test and 19 percent with the Eijkman test. Though neither test proved entirely satisfactory, the Koser test appears to be the better of the two, and further work should be done to determine whether it can be adapted for testing of orange juice.

High Frequency Heating at 30 Megacycles

The specific conductivities of a number of fruits and vegetables were measured at room temperature (25°C.) at various frequencies between 10^7 and 4×10^7 cycles per second. The specific conductivities of fruits at 30 megacycles are about a thousand-fold greater than those of other biological materials commonly heated by the dielectric heating method (for example, wood). These conductivities are so large that it is difficult, if not impossible, to obtain reasonable heating rates at permissible radio frequency voltages. Large heating rates can be obtained at 30 megacycles only by the use of rf voltages so large as to cause arcing between adjacent fruit pieces.

Corrosion of Stainless Steel Equipment

Corrosion resistance of stainless steel processing equipment can be determined with certainty only in use, since corrosion has been found under conditions not ordinarily considered corrosive. The evidence obtained indicates that types 304 and 316 stainless steels are subject to differential aeration corrosion in the presence of a moist sulphur dioxide-air atmosphere. If organic material is being processed, thorough cleaning of the entire system between runs and at the end of each period of operation is imperative. The system should be drained and dry when not in use.

DEHYDRATION

Distribution Costs for Dehydrofrozen Apples

In order to compare the economic position of dehydrofrozen apples with that of apples frozen in the usual way, estimates were made of the various costs incurred in getting the product to the consumer. The consumer, in this study, was assumed to be the institutional trade purchasing at the wholesale level. The costs considered were raw material, processing, storage, transportation, distribution, and brokerage. This channel is common in the marketing of frozen foods. The cost of processing dehydrofrozen apples was estimated at \$0.0532 per pound as compared with \$0.0402 for the frozen equivalent (2 pounds). Offsetting this cost disadvantage were factors favorable to dehydrofrozen apples such as packaging, freezing, storage, and transportation. After evaluation of these steps, it is estimated that dehydrofrozen apples could reach the institutional buyer at a saving of approximately 20 percent as compared with frozen apples.

Boiling on Heated Surfaces

A technique has been developed which will permit the determination of temperature to within 0.01°C. in the study of boiling from heated surfaces in the temperature range of 0° to 140°C.

Spray Drying

While the large spray drier has been inactive awaiting altera-

tions, drying tests have been made with the plasma spray drier on feeds composed of varying amounts of Boysenberry pulp, milk solids, and pectin. These tests indicate that a satisfactory powder (judged on the basis of the "sticky point" moisture content relationships) cannot be obtained when the ratio of milk solids to Boysenberry pulp solids is appreciably less than 1:1. With the powder resulting from a feed composed of 50 percent milk solids and 50 percent Boysenberry pulp solids, a typical ice cream was prepared and compared with an ice cream of identical composition prepared from some of the original Boysenberry pulp before drying. With the triangle plan and a taste panel of about 15, the results indicate that no significant deterioration in flavor had occurred during the drying step.

Pilot Plant Study

The pectin pilot plant equipment, formerly located at Chino, California has been moved to the Western Regional Research Laboratory under an informal memorandum of understanding with Libby, McNeill and Libby. The countercurrent extractor and the vacuum evaporative cooler have been altered at a fabricating plant in Los Angeles and shipped to the Laboratory. Installation of the equipment has been completed in the new location. Operations on washed, dried orange peel are expected to get under way during the spring months.

PECTIN

Pectin Analysis

The accuracy of the optical rotation method for pectin is limited and should be determined from each source until the uniformity of its structure is established. An approximate value for citrus is 235° and for apples is 230° .

Nature and Occurrence of Pectic Substances

Isolation and characterization of pectin in fruits and vegetables is necessary to provide information for the maximum utilization of the optical rotation method of analysis, in order to evaluate such materials as sources of pectin, and also for information useful in the processing field. The plan of such a program is as follows: pectin is extracted by means of slightly acid solutions containing a calcium-sequestering agent; it is isolated and purified under conditions leading to a maximum retention of jelly strength and then it is characterized according to methoxyl and uronic acid content, intrinsic viscosity, osmotic pressure, jelly grade, and specific rotation. Pectins have been obtained from apricots, apples, pea pods, peaches, pears, carrots, potatoes, tomatoes, and sugar beets and their characterization is in progress.

Potential Uses of Pectic Substances

Pectinate Films: Pectinate films are readily applied to a variety of materials to produce a transparent, glossy, nonsticky and protective covering. Methods of drying the film and rendering it resistant to certain microorganisms are being investigated. It

has been found that an air circulation oven will dry pectinate films on candied fruit in eight minutes at 80° C. This appears to be an optimal value for the apparatus used. The optimal moisture content of the films on candied fruit is about 35 percent. Attempts to improve the resistance to fungal attack by use of propylene oxide, Palstone, and other agents are being investigated.

The film containing seed disinfectants has been applied to hybrid corn seed in an effort to increase the percentage germination of seed corn when exposed to adverse conditions. The experiment is still in progress although it may be noted that the film can be applied provided small percentages of glycerol and a wetting agent are employed in the pectinate solution.

A possible use for pectinate films is on frozen food where the water content of the film would be kept as high as possible. Rate of diffusion of gases through a gel film will be controlled primarily by the rate of solution in water while dehydration of the product will be slight until most of the water has been removed from the pectinate gel. Some experiments were tried with round steak as the test meat. When a low-viscosity pectinate is used in a 3.5 solution, the resulting gel loses water at such a rate that the coating would be dry in two months. It is possible to apply a thicker gel by increasing the concentration or using a higher viscosity pectinate or by adding a small amount of calcium ion to the pectinate solution in order to increase its viscosity. Another method is to add agents which will decrease the rate of evaporation of water from the surface of the gel. This latter procedure is now being investigated because of its importance not only to the gel but also the film study.

Other uses of pectinate films depend upon its permeability to water vapor and to oxygen and carbon dioxide. Preliminary results obtained with a relative humidity difference of 50 percent (80 percent on one side of the film and 30 percent on the other side) and a film area of 20 cm² show a permeability of between 30 and 40 mg. of water per sq. cm. per mm. difference in partial pressure of water vapor per hour for a thickness of one micron. Untreated cellophane has a permeability of about 20 in the same units. Further examination of the factors influencing the permeability constant is under way.

Pectinate Gels: Low-sugar gels prepared with low-methoxyl pectin show promise in the dessert and salad field, but further information on their storage characteristics is necessary. A comparative study of gels prepared with samples of low-methoxyl pectin from this Laboratory and from two commercial concerns has been started. The gels are stored at 1°, 25°, and 35° C. Shear modulus and syneresis of the gels have been measured. During the first two weeks an increase in shear modulus was observed but there has been no further change in three months. Syneresis is negligible in all samples. There has been no change in the intrinsic viscosity of the isolated pectin. The storage test is to be continued for nine more months.

Fibers: Fibers of calcium salts of enzymatically prepared low-methoxyl pectins have been spun and tested for strength and elongation. Tenacities of 2 to 3 g. per denier have been attained with extensibilities of 4.6-9 percent. The best yarn had a tenacity of 2.9 g./d. and extensibility of 8.8 percent. These values approach those for cotton, which means that they show promise of being woven in cotton textile machinery as scaffolding fibers.

ASCORBIC ACID FROM WALNUT HULLS

During extraction studies in 1947, about 3,200 gallons of extract were shipped to the Laboratory for concentration and storage for further recovery studies. The extract was clarified in a centrifugal and concentrated 12.4-fold under vacuum. The raw extract had an average concentration of 1,680 ppm. of ascorbic acid; the concentrate had an average ascorbic acid content of 20,600 ppm. The concentration step was accomplished without appreciable loss of vitamin C.

The capital charges on the investment in plant and equipment for the extraction of walnut hulls have been estimated to be approximately \$3.00 per pound of ascorbic acid. The basis for estimates on costs of production have been information supplied by the California Walnut Growers Association on size and location of hullers and also the experimental extraction operations, formerly at Linden and during the 1947 season at Gustine, California, in cooperation with the Association.

Operating costs and the cost of preparing the pure ascorbic acid from the extract liquor would, however, bring the overall cost considerably higher than the \$3.00 per pound for capital charges. On the other hand, some decrease in this cost might be expected if satisfactory methods for storing the hulls were developed so that the processing season could be extended. In view of the high capital charges against the product, as made by the most economical embodiment of the process in full-scale plant operation that can now be visualized, commercial application of the process appears to be a questionable venture in competition with synthetic ascorbic acid.

PROTEIN- AND VITAMIN-RICH FOOD OR FEED SUPPLEMENT

Torula Yeast Investigations

The yeast pilot plant at the Olympia Canning Company was not operated this season. Emphasis has been shifted to the pear-juice separation and fermenter aeration aspects of the problem. With the laboratory pressing data as a guide, a juice separating element was constructed and combined with portions of a Milton-Roy pump and auxiliary valving to form a juice separating machine. The unit was tested at the canning plant during the pear season but gave inconclusive results due to mechanical difficulties. The work is being continued at the Western Regional Research Laboratory.

The laboratory studies on the pressing of milled pear waste have been extended to cover a temperature range of 60 to 180° F. The juice is much more easily separated at elevated temperatures. For a given juice recovery, the pressure and/or draining time required decreases rapidly as the temperature is increased, other factors remaining constant. If pear waste is preheated before pressing, the design and size of juice separating equipment for a given capacity will be substantially different from that required if the waste is pressed cold.

CITRUS

Ethylene Control Experiments for Citrus

Field tests indicate that the portable instrument developed for the estimation of ethylene in the atmospheres of citrus coloring sheds will probably be useful in determining ethylene in air at concentrations down to a few parts per million. Current practice in coloring involves a concentration of about 10 ppm. or more. Field tests with navel oranges undergoing normal coloring treatment indicate that there is no interference due to volatile substances arising from the fruit.

ISOLATION; ANALYSIS, AND CHARACTERIZATION OF CONSTITUENTS

Further work is being done on the sucrose-water system at low temperatures. An investigation of phase equilibria in solutions of sucrose at low temperatures has revealed the existence of at least four crystalline hydrates of sucrose. The cauliflower-like appearance of one of these is responsible for the unsightly "mold-like" growths sometimes found in frozen fruit packed in sucrose sirup. The failure to obtain eutectic mixtures of ice and anhydrous sucrose indicates that formation of this eutectic is probably not a factor of practical importance in frozen food preservation. Work is continuing on low temperature phase equilibria in solutions of sucrose and other sugars.

POULTRY UTILIZATION INVESTIGATIONS

DEHYDRATED EGG STUDIES

Lipid Deterioration

Additional information relative to the mechanism of deterioration of dried egg powders has been obtained. Previous results on the inhibitory effect of phospholipids on development of rancidity in egg fat have been confirmed with more highly purified phospholipid fractions. It appears that aldehydes are formed from the non-phospholipid fraction of the egg fat even in the presence of phospholipids. The aldehydes, however, combine with the reactive groups of the phospholipids to form fluorescing substances, and the egg fat does not become rancid as judged by the usual rancidity criteria. The mode of action of the phospholipids as "antioxidants" for lipids therefore seems to be other

than merely as preferential oxygen acceptors. Experiments to further elucidate this question are in progress.

Preliminary experiments indicate the feasibility of obtaining typical egg components labeled with isotopic tracer atoms by feeding labeled simple glycerides or other carbon compounds to the hens. Studies with labeled egg components should result in more complete identification of the interaction of the egg components responsible for deterioration.

The results of a comprehensive study have shown that egg enzymes are not important in the changes that are responsible for quality loss in processed eggs. It would appear therefore, that efforts to control the deteriorative reactions in dried eggs should concern nonenzymic factors.

FREEZING PRESERVATION

Effect of Alfalfa Meal in the Diet on the Stability of Frozen Eviscerated Turkeys

As a result of feed shortages it has appeared desirable to increase the amount of alfalfa meal in the diets of turkeys. The question was raised whether turkeys fed a high alfalfa-meal diet would hold up well in frozen storage. This report concerns the experimental storage of turkeys fed diets containing 15, 20, 25, 30, 35, 40, and 50 percent alfalfa meal. The turkeys were fed and killed at the Utah State Agricultural Experiment Station, Logan, Utah. They were New York dressed, frozen, transported to this Laboratory, thawed, eviscerated, packaged in heavy waxed paper, refrozen, and stored at -10° F. After 10 months in storage the following determinations were made on two birds from each of the six alfalfa feeding levels: palatability, shear force, cooking loss, peroxide value, Kreis test value, and carotene content. In no case was there a significant trend in the factors measured. Hence, the frozen storage stability of turkeys was not altered significantly by increasing the alfalfa meal in the diet from 15 to 50 percent. This conclusion should be tempered by the observations that pronounced gall bladder discoloration occurred in ten of the twelve birds used in the experiment and that an undesirable odor was associated with the uncooked birds. These conditions were sufficiently pronounced that small differences in the flavor of the meat due to the alfalfa feed could have been masked.

FIBERS, PLASTICS, AND ADHESIVES FROM EGG AND FEATHER (KERATIN) PROTEINS

Solubilization of Keratin Proteins

Procedures for solubilizing feathers with the ethyl ether of ethylene glycol in place of methyl and ethyl alcohols have been worked out. The former offers distinct advantages over the

lower alcohols: first, it forms an azeotrope with water which is directly suitable for solubilization of the keratin; second, it is nontoxic, as compared with methyl alcohol; third, it is not subject to restrictions in use as is the case with ethyl alcohol; and fourth, it has a higher boiling point, permitting a higher temperature for the processing of the keratin and thus reducing the time required for solubilization from 2-1/4 hours to less than 1/2 hour. The fibers spun from the keratin prepared with the ethyl ether of ethylene glycol appear to have as desirable qualities as those prepared from the lower alcohols.

The serine content of feather keratin is high, suggesting that feathers constitute a possible source of this amino acid. The feather keratin prepared by the process described is digestible by proteolytic enzymes. A new design for a screw feed for the extrusion of the keratin as bristles has been developed. Comprehensive theoretical studies have been carried out on the interactions of keratin and alcohols.

ISOLATION, ANALYSIS, AND CHARACTERIZATION OF CONSTITUENTS

Trypsin Inhibitor from Egg White

In confirmation of recent reports, the trypsin inhibitor of egg white would not appear to be potentially useful as an antibiotic. The results on the preparation of derivatives of ovomucoid and trypsin and on the interaction of ovomucoid and trypsin are of significance chiefly because they provide information regarding the fundamental mechanism of specific interaction of large molecules. Such inter-actions are encountered not only in enzyme chemistry but also in the field of toxin and antitoxin reactions, and in the field of antigen-antibody reactions in general.

X-ray Diffraction Investigation of Lysozyme Chloride:

Two crystalline modifications of lysozyme chloride have been grown. A tetragonal form grows at pH 4.5 and an orthorhombic form grows at pH's in the neighborhood of 10. Complete oscillation photographs have been taken of both air-dried and wet tetragonal crystals and of wet orthorhombic crystals. The X-ray data from the air-dried crystals, combined with the density as determined in ethylene bromide and xylene mixtures, has been used to calculate the molecular weight of lysozyme. The value found is 13,900. \pm 500.

VEGETABLE UTILIZATION INVESTIGATIONS

DEHYDRATION

Theory of Drying

Study of the damage to quality which occurs during dehydration have been handicapped by our inability to get inside

a piece while it is drying to see what the conditions are there at any moment. Moisture analyses take a general average of conditions throughout the piece. In the absence of any good experimental method of "getting inside", use has been made in a preliminary way of the theory of diffusion. The results are promising enough to warrant further work along this line. The mathematical work is extremely laborious and slow, however. It is hoped that the rapid electronic computer now being built in California can be used in an extension of this study.

Moisture Determination

A new and accurate method has been developed for moisture determination of dehydrated vegetables. The method consists of allowing the sample to absorb a large quantity of water to swell the tissues, followed by freezing to -70°C , drying from the frozen state, and completion of the drying in a vacuum oven or over a desiccant. This method will be useful as a reference method for the calibration of other more rapid but empirical procedures used in industrial laboratories. It will probably also find application in moisture determinations of other food products.

Storage Studies

Generalizations regarding the effects of temperature, moisture content, and atmosphere of package, upon the rate of sulfite disappearance in dehydrated carrot, white potato, and cabbage have been adduced from the data of the comprehensive storage studies. On the basis of these results, it is feasible to estimate the life expectancy of the sulfite in other samples of dehydrated vegetables. Such predictions are of importance to the vegetable dehydration industry, not only because the inhibitory effect of sulfite upon browning of dehydrated vegetables declines with disappearance of the sulfite, but also because of the unpalatability of dehydrated vegetables in which too much sulfite is retained.

Browning Reaction

The very high temperature coefficient of the browning reaction emphasizes the need of storing dehydrated foods at as low a temperature as possible to avoid undesirable discoloration. Other observations are of importance in throwing light on the mechanism of the browning reaction. This project is being discontinued.

FREEZING PRESERVATION

Frozen and Dehydrofrozen Peas

The results adduced relative to processing and storage investigations on peas, frozen and dehydrofrozen, emphasize the need of improvements in the technology of manufacture. Moreover, they

indicate that a major source of the objectionable off-flavors, frequently found in samples of commercial frozen peas, lies in excessive time-delay between harvest and blanch. The results indicate that (1) solution of this problem lies in (a) rapid flow of raw material or (b) change in method of harvest so that any appreciable time-delay is applied to refrigerated "peas-in-pod", (2) refrigeration of the shelled pea is a palliative and not a solution to the problem.

The indicated lower "air storage" stability of dehydro-frozen peas, compared to conventionally frozen, emphasize the need for further work for the purpose of verification and elucidation of the factors responsible for the difference. The high degree of organoleptic quality retained by dehydro-frozen peas, stored one year at -10° F., encourages continued work on the development of this new method of preservation.

Effect of Freezing Rate on Quality of Frozen Asparagus

The texture and flavor of frozen asparagus appears to be dependent on the rate of freezing, the more rapid rates giving the higher quality. Very rapid freezing, however, may result in shattering of the pieces.

Freezing Preservation of Sliced Potatoes

Small quantities of sliced potatoes of two varieties, Florida Triumph and Klamath Russet, have been prepared and frozen to be subsequently used for deep fat frying. The potatoes were classified on the basis of brine separation. Those of the Florida Triumph variety that sank in an 8 percent brine and Klamath Russet sinkers in an 11 percent brine were used for freezing. A portion of each lot was sliced to 3/16 inch thickness and the remainder cut into Julienne strips. It was found that a 40-second steam scald and a 30-second water scald at approximately 212° F. were sufficient to give a negative peroxidase test with the Julienne strips and 50 seconds in the water and one minute in steam were sufficient for the slices. The slices and Julienne strips, frozen and stored at -10° F., are being fried in deep fat and appraised.

Botulinum Toxin Tests

Examination of the experimental frozen packs of peas, green lima beans, and asparagus, partly inoculated with spores of Clostridium botulinum, has continued. The results are in line with those previously noted; the vegetables, even when inoculated, do not become toxic when held at room temperature for two days. Only when special bacteriological technique is employed can toxic cultures be obtained. With the exception of four cultures from commercially packed spinach and two cultures from lima beans, Cl. botulinum has not been isolated from uninoculated material. About 600 samples have now been examined, 400 for toxin after two days standing at 75° F., and 200 for the

presence of botulinum spores. There is no evidence that the hermetic container would introduce an element of danger from the public health viewpoint when used for frozen vegetables. In only a few instances could inoculated asparagus be made to yield a toxic culture. It was thought that its content of rutin might be responsible. Rutin was not found responsible, but it was found that quercetin in amounts of 100-160 ppm. inhibited growth of Cl. botulinum. In repeated trials, with these concentrations, cultures remained clear and did not become toxic. Quercitrin, on the other hand, had only a feeble effect.

Low Temperature Hygrometry

A new type of dew-point apparatus suitable for use at subfreezing temperatures has been invented and a patent application is being prepared. In the conventional apparatus a mirror is cooled and the temperature at which a fog just begins to form upon its surface is taken as the dew-point. At the temperatures used in freezing preservation the mass of water per unit volume of air is so small that the mirror must be held below the dew-point for an appreciable time before a detectable fog (frost) forms. This introduces a lag which makes it difficult to determine the dew-point precisely. In the apparatus under development a static, linear temperature gradient is established along a polished rod. As air is passed across the surface, moisture condenses on the portion which is below the dewpoint and a sharp boundary is formed which can be located with precision. The temperature at the boundary is obtained by interpolation between two points on the rod where the temperatures are measured by means of imbedded resistance thermometer elements.

Improved Calorimeter

A calorimeter for determining the amount of heat removed from sample packages during freezing has been developed. By maintaining the bath temperature constant at approximately room temperature it was possible to virtually eliminate heat losses through the calorimeter. The usual practice of computing the water equivalent of the calorimeter is also eliminated by preheating the calorimeter before introducing the samples. Calorimetric determinations on one-pound samples of ice in an experimental model were accurate to within 1 percent.

Utilities Usage in Frozen Food Plants

Data gathered in cooperation with the Bonneville Power Administration were analyzed and ratios of electric consumption per unit of product for existing plants were determined by examining the range, median, and average obtained from the array of "interview and check lists," one of these having been prepared in the field for each plant visited. These ratios were then increased (based on estimates, prepared by the Laboratory staff) to form a basis for forecasting future electrical utilization. The Bonneville Power Administration will use the ratios in preparing

its estimate of power requirements for the Army Engineers' Columbia Basin Review Report. A copy of the report and duplicates of the check lists have been retained by the Laboratory. In addition to the electrical data these check lists contain actual figures on other plant operating costs, such as labor, steam, other fuels and inplant generation of electricity.

Cooperative Freezer Tests

Field tests were conducted by members of the Laboratory staff at the Security Warehouse and Cold Storage Company, San Jose, California for the purpose of securing engineering and operating data for commercial air-blast freezers. The temperature measurements showed that the time required for 12-ounce packages to attain a center temperature of 0° F. varied from 4-1/2 to 8-3/4 hours at different locations within the freezer. During the test period, the air entered the freezer at an average temperature of -8° F.

Uneven air distribution was responsible in part for the wide range of freezing rates. Observed air velocities over the test packages ranged from about 10 ft. to 580 ft./min. In general, air velocities were much higher near the floor of the freezing section than near the ceiling, and excessive by-passing of air occurred in the spaces above, below and between the freezer frames. Air velocities ranging up to 1,200 ft./min. were observed between the bottom of the freezer frames and the floor.

Measurement of Air Velocity in Air-Blast Freezers

Experimental work has continued on heated thermocouple anemometers. By means of changes in length of heated junction, a common calibration curve was established for fifteen of the anemometers at room temperature. The same anemometers were recalibrated at -100° F., using the same orifice meter. Theoretically, the data at -100° F., should represent one of a family of temperature curves on a log-log chart. The results, however, were represented by three curves, one for each of the three orifices used. It is apparent that accuracy of the orifice meter was exceeded by that of the anemometers at the lower temperatures. To continue the calibration work, a more precise orifice meter and test section have been designed and built.

Spectrophotometric Investigation of Reflectance Color of Foods

Studies of reflectance color of vegetables are being made in an attempt to correlate objectively measurable color factors with subjective preference. Samples have been graded for preference by a selected panel and simultaneous measurements of color made on the General Electric Recording spectrophotometer and the Hunter Reflectometer. Well established objective methods of color measurement will be used in food appraisal studies.

SUBTILIN

Marked progress has been made in the various phases of work on this antibiotic.

Production

Studies of the nutritional factors that affect the formation of subtilin by Bacillus subtilis and the application of these results to its submerged culturing have permitted a greatly expanded program of subtilin production. It is now being produced on a semipilot plant scale on a sucrose-ammonium citrate medium containing the proper amounts of essential trace elements. The average fermentation yield is about 0.4 gm. per liter of culture. A weekly schedule consists of 3 consecutive runs with fermenter charges of 200, 150 and 200 liters with a production of 200 gm of subtilin, 50 percent (100 gm) of which is ordinarily isolated.

Isolation and Purification

Introduction of a "salting out" step for the precipitation of subtilin from the butanol culture extract permits its recovery as a centrifuge cake, thus effecting its separation from the aqueous butanol phase in a simple operation without the use of hazardous flammable solvents. (The butanol is available for recovery). The final steps of purification are essentially the same as previously reported.

Assay

A batch of highest potency subtilin (L 1373), produced and isolated as described above, has been reserved as a standard preparation. It has a potency of 23% in comparison with the former arbitrary standard lot L 1263.

A cup plate assay method using Sarcina Lutea as the test organism has been adapted for measurement of blood levels from subtilin. This bacterium is considerably more sensitive than Micrococcus conglomeratus, while the yellow pigment aids in measuring the inhibition zones. A 20 mm. zone is obtained with 1.5 ppm. of subtilin if the plates are incubated at once, with 0.4 ppm. if the subtilin is allowed to diffuse for 16 hours in a refrigerator before incubation. Subtilin in citrated blood gives a dosage-response curve with less slope than pure subtilin, but by comparison at an arbitrary zone diameter semiquantitative blood levels have been estimated in connection with investigations of mode of administration of subtilin being made by the Pharmacology Division.

Properties

Subtilin preparations of high potency were shown to be homogeneous with respect to salt fractionation and dialysis. Electrophoretic studies of the dialyzed material support the conclusion

that subtilin is electrophoretically homogeneous. The solubility of subtilin in blood serum or in 0.85% sodium chloride at a pH of 7.4 and 37° C. is 0.06 - 0.07%. Its molecular weight is around 7000.

Chemical Modification

Limited solubility of subtilin in blood serum has led to a program directed at the production of more salt-soluble, anti-biotically active derivatives. Subtilin is a polypeptide characterized by the presence of certain of the common amino acids, the substantial absence of others, and by the presence of lanthionine. The molecule thus offers the possibility of modification by a number of specific protein reagents. Most work has been done thus far with a partially methylated derivative which is characterized by a solubility under physiological conditions increased about two-fold and an antibiotic potency increased more than three-fold. The antibiotic spectrum of this methylated subtilin is unchanged qualitatively.

Evaluation

Improvement in the methods of production and isolation of subtilin have made possible its production in amounts sufficient for distribution of samples to qualified institutions for its evaluation in the treatment of specific diseases. Included in these are the following:

Tuberculosis - Department of Bacteriology, University of California,
Los Angeles

University of California Medical School
San Francisco County Hospital
National Institute of Health
Merck and Company
Squibb Institute for Medical Research

Trichomonas vaginalis - University of California Medical School

Rickettsial diseases - Lederle Research Laboratory

Endamoeba histolytica - University of California Medical School

Bovine mastitis - School of Veterinary Medicine, Washington State
College

Department of Veterinary Science, University
of Minnesota

Bureau of Animal Industry, Beltsville, Maryland

Coccidiosis - Department of Poultry Pathology, University of
California

In addition to the above, a sizable amount of subtilin has been supplied to the Antibiotics Study Section of the National Institute of Health for distribution to qualified research workers, as well as small samples to many other responsible groups without commitments as to the exact nature of the work to be done.

The Pharmacology Division of this Bureau is actively engaged in the study of modes of administration of subtilin and the determination of the toxicity of subtilin and its derivatives.

WHEAT UTILIZATION INVESTIGATIONS

INDUSTRIAL NONFOOD USES OF GLUTEN

Phosphorylation of Proteins

Studies on the reactions occurring when wheat proteins are treated with phosphoric acid containing excess phosphorus pentoxide have been completed. The same reactions do not occur with "phospholeum", a commercial phosphoric acid containing excess anhydride. The study of protein phosphorylation is being discontinued. No advantages over sulfation for the preparation of gel-forming products have been discovered.

GLUTEN SULFATE

Sulfation with Sulfur Trioxide

The highly corrosive properties and instability of commercial sulfur trioxide have slowed progress in the investigation of its possible use for making gluten sulfate. Glutens prepared from soft white wheats have been sulfated by the standard sulfuric acid procedure. One preparation had a hydration capacity of over 500, that is, a firm gel was prepared in which the solids content was only 0.2 percent. The possibility that soft wheat glutens might yield sulfated products of greater hydration capacity than hitherto observed is suggested by these preliminary observations.

ISOLATION, ANALYSIS AND CHARACTERIZATION OF CONSTITUENTS

Lipid-Protein Relationships in Gluten

Despite the large number of analyses that have been run, no essential difference has as yet been detected between flours of equal protein content that are known to differ markedly in baking properties. This conclusion is disappointing; nevertheless, a considerable amount of information has accumulated that may, at a later date, prove valuable.

PROJECTS UNDER THE RESEARCH AND MARKETING ACT OF 1946

The Western Regional Research Laboratory has been assigned projects under this act on the following subjects:

1. Develop New and Improved Uses for Rice and Rice By-products and Better Methods of Processing and Handling Prior and Subsequent to Processing.

Work under this project will be conducted partly at the Southern Regional Research Laboratory and partly at the Western Regional Research Laboratory. At the Western Laboratory the initial project will be on "Studies in the storage of rice and its component parts."

2. Development of New and Improved Food, Feed, and Industrial Uses for Dried Beans, and Splits and Culls Therefrom.

Under this project studies will be made on "Factors affecting the quality of processed dried beans and peas" and "Survey of industrial utilization of dried peas and beans."

3. Quality Preservation of Poultry Products, Improved Products and Improved Techniques of Maintaining Quality in Storage.

Under this project the first subject to be investigated will be "Freezing preservation of cooked turkey meat."

4. New and Improved Uses of Deciduous Fruits Especially to Prevent Waste.

Under this project the first subjects to be studied will be "Development of processes and equipment for the production of deciduous fruit concentrates" and "Recovery and utilization of deciduous fruit essences and concentrates."

5. New and Improved Uses of Citrus Products.

Under this project subjects to be investigated include the "Development of Procedures for large scale production of citrus fruit powders" and "Development and utilization of citrus powders."

6. Study of Fundamental Characteristics of Wool as a Means of Developing New and Improved Uses.

Under this project the subjects to be studied include "Physical investigations of natural and modified wool," "Modification of wool by chemical means" and "Mechanical and molecular-kinetic characterization of natural and modified wools."

7. Submerged Fermentation Production of Edible Mushroom Mycelium from Agricultural Wastes.

Under this project the subject to be studied will be "Production of edible mushroom products by vat fermentation."

8. A. Conversion of Potatoes to More Stable Forms and Products.
- B. New Methods of Using Potatoes for Feeds and Non-Food Uses.

Work under this project will include "Utilization of raw, dehydrated and dehydrofrozen white potato for the manufacture of potato chips and related products" and "Development of means of stabilizing peeled potatoes intended for shortly delayed use and possibly as a packaged product for household use."

Another project which it is planned to conduct under a contract administered through this laboratory is to "Design and Construct Commercial Model of Continuous Type Press for Recovery of Juice from Pears and Other Fruits and Their Wastes Presenting Similar Juicing Problems."

Other projects have been and are being considered, and it is probable that some additions will be made to the list.

The work under the above projects has not progressed far enough to warrant a report on accomplishments at this time. Efforts thus far have been devoted largely towards assembling a staff with appropriate equipment and quarters to conduct the work. Approximately 35 professional workers are being added to our staff for these projects.

EASTERN REGIONAL RESEARCH LABORATORY
P. A. Wells, Director

APPLE AND OTHER FRUIT UTILIZATION INVESTIGATIONS

JUICE PRODUCTS

Preparation and Storage of Apple Juice

The experiments reported last year on processing and storage of apple juice were repeated in the 1946-47 season. However, in order to determine the effect of apple storage on juice quality, additional samples of juice were prepared under the same conditions, from the same lot of apples after three months' cold storage. Each lot was divided and half stored at 35° F. and half at 75° F. These were tested at six-week intervals by a taste panel. The results of the storage tests showed that juice held at 35° F. for 12 months retained most of the original flavor, whereas juice stored at 75° F. consistently rated two or three points (scale 10 to 1) poorer in flavor. These results confirm the previous season's study.

A definite preference for cloudy juice in contrast to clarified juice was shown at the cold storage level; however, at room temperature storage they were about equal in quality. All the cloudy juices contained sediment, whereas the clarified juices had very little or none. Cold storage seemed to retard sediment in the latter case.

The juices packed from apples kept in cold storage for three months were definitely inferior to the juices prepared from fresh apples and then stored three months in cold storage. Addition of apple essence improved the flavor of both clarified and cloudy juices and retained this improvement throughout the year.

Apple Juice Survey

During May and June a survey was made of the commercial apple juice packed in 1946. Twenty-nine producers submitted 36 samples of juice representing a total production of 16,000,000 gallons, or 80 percent of the probable total pack. This represents a considerable increase in production over the 5,850,000 gallons packed in 1941, the date of the last previous survey.

The survey indicated a trend away from deaeration, and a trend toward the addition of ascorbic acid. However, there were not sufficient samples of ascorbic acid fortified juice to indicate to what extent, if any, the juice was improved by this treatment. The average flavor score of the samples submitted was lower than that observed for the 1940 and 1941 surveys. The fact that the 1946 survey was conducted about 2 months later in the season than the previous surveys may account in part for the poorer flavor scores, since it has been observed that flavor deteriorates on storage, especially at room temperatures. Laboratory lots of juice stored 7 months at 75° F. averaged two flavor grades lower than those stored at 35° F.

Such factors as the type of container, deaeration, use of ascorbic acid, filtration, clear or cloudy type of juice, and size of plant showed no apparent relation to flavor score.

Because the survey showed that half of the commercial brands of juice rated poor or objectionable, and since the marketing of juice of such inferior quality undoubtedly hurts the industry as a whole, it was decided to visit some of the apple juice plants and observe their operations. Twenty plants in Virginia, Pennsylvania, Ohio, Michigan and New York were visited. Of this number, 12 were in actual operation, 3 had not yet started production this season, and 5 had discontinued apple juice either for this season or permanently. Of the 15 plants producing or expecting to produce juice this season, only 7 have any technically trained personnel to supervise quality control. Two or 3 others have the services of outside consultants and at least 5 have no scientifically trained assistance of any kind. This condition of quality control, or lack of it, is usually reflected in the quality of product from the individual plants.

As a general rule apple juice is considered a salvage operation and little attention is given to quality of raw material. Many plants were using a large proportion of small, immature fruit which was low in sugar and had very little flavor. The inspection of fruit to eliminate rots and bruises was generally inadequate and at one plant there was no inspection at all.

The sanitary conditions at the various plants varied from fairly good to extremely poor. At several plants the improper cleaning of press cloths, tanks, and other equipment actually imparted off-flavors to the juice. Even the best managed plants do not approach the cleanliness of grape juice plants.

There is a general lack of appreciation of the harmful effects of high temperature storage. Only one or two plants cooled the juice adequately before storage. At most plants the juice was cooled only to 115-120° F. (136° F. at one plant) and immediately stored in tight stacks with little chance for further cooling. Such juice often develops off-flavors within a week.

As a consequence of the above findings, we have temporarily reversed our viewpoint in the study of apple juice. We are now seeking the causes of poor juice. We are getting experimental evidence on the effects of warm storage, decayed fruit, dirty press cloths, immature fruit and other supposedly ulterior factors.

Volatile Flavor Recovery

During the past year a small portable flavor recovery unit was constructed. Essences were produced from a variety of fruits, with this equipment, in cooperation with the Horticulture Department of the Virginia State Experiment Station. The unit was operated at Blacksburg, Virginia by members of the Eastern Laboratory staff, on juices prepared by Dr. L. L. Davis from frozen fruits and juices which he had collected for us during the spring and summer. Juices from strawberries, peaches, blackberries, youngberries, rhubarb,

huckleberries, Delaware grapes, Moore's Early grapes, Niagara grapes, Worden grapes and two varieties of French cider apples were processed.

The essences from strawberries, peaches, huckleberries, rhubarb, Moore's Early grapes and Worden grapes had strong characteristic aroma. Niagara grape and Delaware grape essences were strong and fragrant, but lacked full grape character. Blackberry and youngberry essences were less characteristic of the full berry flavors, but contained the top note and in concentrated form are recognizable. Essence produced from juice expressed from Moore's Early grape pulp is not characteristically grape, but essence produced from juice expressed from the skins of this grape is highly characteristic. This indicates that the grape skin contains the bulk of the volatile aromatic constituents associated with grapes.

The apple essences produced from the two French cider varieties were only fair, but they would not be objectionable in any reasonable blend of apples.

Industrial interest in the production and application of apple and other fruit essences continues at a very active pace. However, the recent imposition of a \$9.00 per wine gallon tax on essences containing over 0.5 percent alcohol is giving some concern. Commercial samples of fruit essences have been sampled that contain up to 10 percent alcohol. A request for a reclassification of these essences has been made by industry as they feel it would make the production of the fruit essences commercially unfeasible.

Firming of Apple Slices

Changes in the firmness of apples stored at 35° F. were measured, using a compression test. Comparing varieties in April, the firmness of cooked apple flesh was in the following decreasing order: eastern Red Delicious, Jonathan, York Imperial, western Red Delicious, Stayman Winesap and McIntosh. The firmness of the eastern Red Delicious, Jonathan and York Imperial remained fairly constant from January until the apples spoiled, whereas a gradual decrease in firmness occurred in the western Red Delicious, Stayman Winesap and McIntosh apples. Jonathan apples showed the best keeping quality, and cooked tissue of satisfactory firmness was obtained from them even in July. McIntosh apples disintegrated into a mush on cooking as early as in March.

The effect of pH on the firmness of cooked Jonathan apples was measured. The firmness decreased markedly at pH below about 3.5 (the natural pH), was almost constant from 3.5 to 7.0, and decreased gradually above pH 7.0. The effectiveness of calcium ion as a firming agent increased almost linearly with pH, approximately doubling with a pH change of one unit.

A number of substances other than calcium ion were tested as firming agents for Jonathan apples. Jonathans were used because at the time when the tests could be begun they alone among the several varieties were sufficiently sound. Aluminum ion was as effective as

calcium, whereas magnesium ion showed no firming action at all on Jonathan, western Red Delicious, and Stayman Winesap apples. Magnesium, aluminum, and calcium would appear to be the only metals whose salts could possibly be acceptable as firming agents for fruit. Sucrose at concentrations of 10 and 30 percent doubled and tripled the firmness of cooked Jonathan apples. Exploratory work showed that tannic acid, formaldehyde, and barium salts were as effective as calcium, and that lead ions caused the greatest increase in firmness of all substances tested. These observations, with the fact that calcium ion does not gel or thicken normal apple pectin extracts, suggest that other constituents of the apple, such as hemicellulose, starch or a derivative, protein, or an oxidized cellulose, may be considered jointly with pectin as possible participants in a firming reaction.

Preliminary tests indicate that the soluble fraction of pectin within apple tissue may increase firmness by reacting with calcium ion. Both with a stored winter variety (Jonathan) and an immature summer variety (Gravenstein), about three times as much pectin, determined by the Carre-Haynes method, was extracted by water at 25° C. from untreated tissue as from tissue which had been impregnated with calcium ion.

VEGETABLE UTILIZATION INVESTIGATIONS

VEGETABLE WASTES

Feeding Trials

The widespread use of carotene by the feed industry depends upon the certain knowledge that, unit for unit, carotene is as efficiently utilized as a source of Vitamin A as the natural vitamin from fish liver oil.

In a preliminary experiment at the Delaware Agricultural Experiment Station in 1945, a broccoli carotene extract was compared with fish liver oil at two levels and the results showed that carotene was as efficiently utilized as fish liver oil as a source of Vitamin A. A second experiment has been carried out under much more carefully controlled conditions and the results confirm the previous ones. In this feeding trial molecularly distilled carotene was compared with a commercially prepared molecularly distilled vitamin A oil at three levels using a basal diet essentially free of vitamin A. Carotene in broccoli was also used in this trial.

The data clearly show that the growth, the feed efficiency, and the mortality of chicks receiving their vitamin A as carotene, either in the form of the leaf meal or as the purified extract, were equal to those of chicks receiving their vitamin A in the form of the distilled fish liver oil. A third series of feeding tests, which included both male and female chicks, was in close agreement with the results above.

Three new feeding trials using vegetable waste products are

currently under way at the Delaware Station. A large scale comparison of carotene and vitamin A, involving 6 pens of 50 laying birds each, has been in progress for approximately two months. No significant differences in laying or hatchability have been observed at this early stage between the groups receiving their vitamin A in the form of carotene as compared to the groups receiving fish liver oil.

Vegetable Leaf Proteins

Extensive analytical data on the amino acid contents of vegetable leaf proteins shows that they contain a well-balanced mixture of the ten most essential amino acids for human or animal nutrition, and that all leaves have about the same amino acid make-up.

RUTIN

Agronomic Studies

Continued investigations in cooperation with the Pennsylvania State College and the Bureau of Plant Industry, Soils and Agricultural Engineering, on four varieties of buckwheat showed that the Tartary and tetraploid varieties are equally superior sources of rutin, both from the standpoint of content and yield per acre, followed closely by emarginatum, while the Japanese proved to be definitely inferior.

On DeKalb soil at Clearfield, Pennsylvania, buckwheat showed the greatest response to fertilizer containing nitrogen. This was apparent in more rapid growth of young plants, with a result that there was a higher yield, calculated to dry weight, a somewhat lower rutin content, and an earlier peak in yield of rutin per acre, than with phosphorus, with phosphorus-potassium, or with lime. After seeds had well developed the dry weight percent and yield of rutin per acre were little affected by fertilizer treatment. These effects were most pronounced with the Tartary variety, which exhibits indeterminant growth, and thus has a more prolonged growing period than the Japanese. An insufficient number of collections were made of the tetraploid and emarginatum (the other two indeterminant varieties) to warrant any definite conclusion on fertilizer response.

These experiments support previous indications that Tartary buckwheat is superior to the Japanese variety as a source of rutin. As a result, virtually all the rutin now being produced comes from the Tartary variety. However, the relationships between rutin yield and fertilization of buckwheat have not yet been completely evaluated.

Manufacture from Buckwheat

The first commercial production of rutin from buckwheat by processes developed under this project occurred in 1945, when four

companies produced about four hundred pounds; in 1946 at least ten companies engaged in rutin manufacture, producing an estimated 10,000 pounds. Due to increased interest in this drug throughout the medical profession, it is believed that manufacturing operations this year are on a somewhat larger scale. Rutin is now available on prescription throughout the United States.

Biological and Clinical Studies

The practice of furnishing rutin to physicians for individual case treatments was, in general, discontinued in September 1946, when the drug was offered for sale to the public. Moreover, since we found that results from isolated cases are of doubtful medical value, except in unusual circumstances, rutin is now being furnished principally to clinics for experimental treatment of a variety of disorders. Among these may be mentioned apoplexy, diabetic retinitis, hemophilia, purpura, arteriosclerosis, coronary thrombosis and hypertension. On the basis of the known effect of the drug on capillary fragility and permeability, these disorders may respond favorably to rutin therapy.

The use of rutin in preventing damage from frostbite in rabbits has been investigated extensively during the past year at Stanford University Medical School. The results show that rutin is invaluable in preventing frostbite injuries. The possible military implications of this discovery are apparent, and an effort will be made this year to initiate clinical testing under field conditions. In a previous report it was indicated that studies by Dr. J. Q. Griffith, Jr., of the University of Pennsylvania Medical School, showed the value of rutin in preventing X-ray burns. It was conceived on the basis of these results that rutin might be useful in alleviating some of the known injurious effects of atomic radiation. Studies to test this hypothesis are now being carried on in cooperation with laboratories of the Atomic Energy Commission.

Considerable progress was made in laboratory studies to develop suitable chemical and biological methods for assaying rutin. One method developed by this Bureau's Pharmacology Division is based on the effect of rutin on capillary permeability. When this technique is developed to give accurate quantitative results it may afford valuable information on the mechanism of rutin action. So far we have no basis for explaining the important effects of this substance.

TOBACCO UTILIZATION INVESTIGATIONS

Tobacco Conference

In accordance with suggestions made at the Experiment Station Directors' Meeting in Philadelphia last May, a conference on tobacco research was held at the Eastern Laboratory on October 22 and 23. Those attending included most of our collaborators, and representatives from Universities, the tobacco industry and interested Department agencies. A summary report of the conference was made available to Directors of all State Experiment Stations.

Activators for Nicotine

For the past several years attempts have been made to improve the usefulness of nicotine as an insecticide by finding a suitable activator or synergist. Synergism has been appropriately defined as the property that makes "two plus two equal five," i.e., the effectiveness of the combination is greater than the additive effect of the materials taken singly. Since there is no basis for predicting synergistic effects, the substances chosen for testing were picked at random. Several hundred compounds were tested before one was found which showed a definite activating effect. This substance, phthalonitrile, has been carefully studied during the past year. Under the conditions used, mixtures of phthalonitrile and nicotine showed marked synergism against the armyworm, definite synergism against the pea aphid, diamondback moth, and California oak worm, doubtful synergism against the green dockbeetle and celery leaf tier, and no synergism against the pomace fly and greenhouse thrip. Synergism was shown by three forms of nicotine -- sulfate, bentonite, and cuprous nicotine cyanide -- as a stomach poison and fumigant. In many cases the toxicity of the nicotine was doubled or trebled. Although phthalonitrile may not prove to be a feasible adjunct to be used with nicotine, it does prove that synergism with nicotine is possible. The search for others can continue with increased confidence.

NICOTIANA RUSTICA

Cooperative Agronomic Studies

The informal cooperative arrangement with the Bureau of Plant Industry, Soils, and Agricultural Engineering has been continued for the growing of plant material, and for studies on nicotine production. Plots were continued at Wyndmoor, Pennsylvania; Beltsville, Maryland; Waynesville and Transou, North Carolina; and at the Alaska Experiment Station. An additional plot was grown at Highlands, North Carolina, in an area of high rainfall and fog.

The production of the 1947 plots was regarded as normal. In Alaska a short season German variety of rustica was tested, and first reports indicate that it will prove to be a better selection than the larger, slower maturing Department varieties used in 1946.

The new selections developed by the Department at Arlington Farm and Beltsville, some by Dr. H. H. Smith, 12 varieties secured from Russia, and one German variety were also included. The Department selections are large growing and possess maximum sucker growth, a costly economic factor. The Russian and German selections are smaller plants, with a lower suckering cost. Some of them showed possibilities for use in the humid regions like Pennsylvania.

Recovery of Nicotine

Five methods are indicated for preserving the nicotine in

rustica. They are (1) extraction of the juice by pressing in a cane mill or Davenport press and extracting the nicotine from the juice immediately or from storage; storage of the green plant material as (2) ensilage or (3) in acid or alkali solutions; (4) drying the plant material rapidly by artificial heat; (5) natural air drying.

The natural air drying is the obvious method for the farm installation. Like ordinary tobacco the grower can bring his rustica to a safe handling condition. This would allow him to move it to the extraction plant at his convenience. It would require the grower to supply a suitable barn for the drying or curing operation. The other methods of preserving materials listed above require special storage at the extraction works.

In 1946 two ensilage experiments were undertaken. One employed 5 percent molasses to increase the carbohydrate content, and in the second 2 percent phosphoric acid was added to preserve the material. There also was a series of high acid solutions, ranging from pH 1 to pH 4. The tobacco was cut and packed with only a normal wilt. The high acid solutions preserved the nicotine remarkably well with a minimum of loss.

To secure better ensiling conditions in 1947 the tobacco was severely wilted in the field, losing 25 percent of its freshly cut weight, before being packed in the storage jars. The 5 percent sugar solution and 2 percent phosphoric acid ensilage experiments were repeated. Seven solution experiments are also being studied using 3 percent and 5 percent NaCl, 5 percent KCl, 5 percent and 10 percent lime, and 1 percent bentonite. Results of these experiments are not yet available.

Pilot plant scale experiments were made on expressing the juice from green Nicotiana rustica, with the object of eliminating the necessity of steam distillation of the fresh or dried plant to obtain the nicotine. The plants were chopped by an ensilage cutter, then passed five times through a small sugar-cane mill. Before the fourth pass water was added, and again before the fifth. Of the total nicotine in the plants, 92 percent was found in the expressed juice. The concentration of nicotine in the juice from the first pass was about 0.50 percent; the additions of water on the last two passes reduced the concentration in the total mixed juice to 0.45 percent.

Since it is necessary eventually to add lime to the expressed juice in order to prevent fermentation and to liberate the nicotine, tests were made with the addition of 0.8 percent of lime to the chopped plant before milling, in an attempt to make the plant tissue more amenable to pressing. The extraction of nicotine was the same, viz. 92 percent.

Similar experiments were made in a Davenport double-cone press of the smallest commercial size. At low speed both with and without lime the same extraction was obtained as with the cane mill.

At maximum speed the capacity was 2.9 times that at low speed, and the extraction only 2 percent less. In a commercial installation four or five mills, or presses, would be placed in tandem and the plant material passed continuously from each to the next, water being sprayed on it during its passage.

MILK PRODUCTS UTILIZATION INVESTIGATIONS

MILK PROTEIN STUDIES

Bristles from Milk Proteins

In the production of casein bristles quinone has been used as a hardening agent since it gave a flexible, black bristle of good durability. Before the development of the continuous method reported last year, bristle hardened with formaldehyde was invariably brittle. Since the development of the continuous method, it has been found that the formaldehyde treated bristle is as flexible and tough as the quinone bristle.

The use of formaldehyde in hardening casein bristle gives an alternative method of hardening, which results in a colorless bristle that may be dyed any desired color. Moreover, the price of formaldehyde as compared with quinone makes the production of formaldehyde bristle more economical.

It was found that the addition of small quantities (0.1 - 0.5%) of heavy metal salts such as mercuric chloride increased the wet and dry strengths of casein bristles approximately 15%. Mercuric chloride was more effective in increasing the tensile strength of bristles than cadmium and zinc chlorides. Under the conditions of making bristles, mercuric chloride breaks the disulfide bonds of casein and produces a decrease in viscosity. It is not likely that these changes are concerned with the improvement of the tensile strength of bristles by mercuric chloride. However, the addition of a small quantity of mercuric chloride in preparing bristles is advantageous since it is not expensive and has the further advantage of destroying proteolysis and inhibiting breakdown by microorganisms.

Structure of Proteins and Protein Fibers

Continued studies on the water absorption of proteins has yielded results of considerable interest. Since the water absorbing power of the peptide groups in proteins has long been the subject of controversy, our studies along this line during the past year were directed at a solution of the problem.

Two long chain polyglycine molecules were synthesized and their vapor phase water absorption isotherms determined. The shorter glycine peptides previously studied have shown that nonhygroscopic amino acids can produce hygroscopic peptides, but it was necessary to employ these longer peptides to show that the peptide groups themselves were capable of acting as sites for the absorption of water.

The peptide group was shown to be responsible for over 50% of the water absorbed by most proteins. This fact is useful in determining the amount of change in water absorbing properties which can be expected by modification of proteins.

The water absorbed per gram of nitrogen in the backbone chain of these polyglycine peptides is almost equal to that absorbed by silk, which is one of the less complicated proteins. This indicates that the peptide group may have the same activity as a site for water absorption in all proteins.

CARBOHYDRATE STUDIES

Plasticizers from Lactic Acid

Softening or plasticizing oils are mixed with many resins and elastomers to increase flexibility and toughness and to facilitate fabrication operations prior to manufacture of the finished articles. These softeners or plasticizers are an important component of plastics and elastomers, and frequently comprise as much as 30 to 45 percent of the finished composition. The plastics industry as it exists today is dependent upon and is in need of better plasticizers; it is anticipated that as much as 200,000,000 pounds of plasticizers, commanding prices as high as \$1.00 per pound, will be required in 1948.

Because of the importance of plasticizers and the huge potential outlet afforded for these materials, lactic acid derivatives having the properties required in plasticizers were prepared and studied. In conducting this study, particular attention was given to the production by economical methods of products suitable for plasticizing vinyl resins and cellulose derivatives, including cellulose acetate and ethyl cellulose. Some of the experimental plasticizers could be purified by distillation, but it was necessary to refine many of the relatively non-volatile products by treatment with activated carbon. The yields were high, and the reagents used to prepare most of the plasticizers are available commercially at low cost. Certain pertinent properties, such as volatility, compatibility with typical commercial resins, water solubility, boiling water stability, and viscosity of the plasticizer were determined. The plasticizers were mixed with a vinyl chloride resin, and the resulting compositions were used to determine tensile strength, elongation, and flexibility. The promising results obtained thus far in the evaluation studies indicate that lactic acid can be converted into many derivatives that are suitable for plasticizing several of the commercial resins. Some of the lactic acid derivatives were approximately as effective as the currently preferred commercial plasticizers. Several industrial organizations have expressed an interest in lactic acid plasticizers and are conducting evaluation and market studies.

ANIMAL FATS AND OILS UTILIZATION INVESTIGATIONS

STABILITY AND STORAGE INVESTIGATIONS

Studies concerned with the synthesis and evaluation of various new compounds as antioxidants (stabilizers) for fats have been continued. Last year it was reported that studies on the preparation of esters of gallic acid by direct synthesis resulted in

greatly improved yields of these compounds. By the improved procedure pure lauryl gallate, an ester having especially favorable properties for antioxidant use, has been prepared in yields of 70-80 percent. Commercial preparation of this product should be relatively simple.

The antioxidant properties of the gallic acid esters were determined by an accelerated aging test and by baking tests. The latter afford information on the power of the antioxidant to protect the fat in baked products. The rapid aging tests indicate that the higher gallate esters are equal to the best of other known antioxidants in stabilizing animal and vegetable fats. Baking tests with piecrust showed that a higher concentration of lauryl gallate is required for effective stabilization of lard for use in piecrust than is required for bulk fats. About 0.05% is desirable for piecrust, whereas as little as 0.01% is effective for bulk lard. In tests with soda crackers, lauryl gallate was relatively ineffective. This is undoubtedly due to the presence of soda. These results further confirm the value of the higher esters of gallic acid as antioxidants for fats and oils, but define certain limitations as to their effectiveness. In general, they are good antioxidants for animal fats and for hydrogenated vegetable oils, and show good carryover into baked piecrust.

SURFACE ACTIVE AGENTS

Emulsifiers for Manufacture of GR-S Synthetic Rubber

Cooperative research previously reported, undertaken with the Rubber Reserve Company in connection with their Soap Development Program for Government Synthetic Rubber, showed that: (a) the polyunsaturated fatty acid components of the tallow soap emulsifiers acted as retarders in the emulsion polymerization of GR-S synthetic rubber; (b) selective hydrogenation of the fat stock eliminated variations in the rate of polymerization; and (c) spectrophotometric methods of analysis for residual polyunsaturation in the soaps were successful as a means of production control. Specifications for the soaps to be used as emulsifiers were accordingly revised by the Rubber Reserve Company and the program calling for hydrogenation was put into operation and proved successful in 1945 and 1946 in the manufacture of GR-S by the batch process.

Solution of this problem made possible a conversion of the synthetic rubber plants to a continuous process. However, early in 1947 it became apparent that in the continuous process unknown retarders were present in the soaps unrelated to their polyunsaturated fatty acid content, producing variations in the rate of conversion to polymer, particularly in the later stages of polymerization.

Soap samples representing emulsifiers of both good and bad characteristics, with data for their performance in plant scale operations, were supplied to the Laboratory by three Rubber Reserve Company plants, with a request from the Office of Rubber Reserve for assistance in solving this new problem.

Spectrophotometric analyses of these soaps showed most of them to be below the specification limit for polyunsaturated fatty acid content. When corrected for these known retarders, wide variations in the data for percent conversion to polymer remained. This indicated the presence of unidentified retarders, presumably in the soaps.

Semi-quantitative spectrographic examination of the ashed soaps for trace metal content revealed unexpectedly large amounts of nickel in many cases. Other metals were absent. Quantitative colorimetric analyses established a range of 0.0 to 10⁴ parts per million of nickel in fourteen different soaps which we examined.

A plot of nickel content of soap versus percent conversion to polymer indicated a linear relationship between nickel content and drop in percent conversion, approximately 8 parts per million corresponding to a drop of 1%.

It was tentatively concluded that nickel, present as an impurity in the soap owing to incomplete removal of fat hydrogenation catalyst, acts as a retarder in the continuous emulsion polymerization process for manufacture of GR-S synthetic rubber. It was suggested that the Rubber Reserve Company test the effect of added nickel, both in the form of finely divided metal (catalyst) and in combined form such as nickel oleate, in bottle scale polymerizations. The results of such tests have not been reported.

PLASTICS, PLASTICIZERS, COATINGS AND OTHER INDUSTRIAL CHEMICALS

The vinyl and furfuryl esters of caproic, caprylic, pelargonic, capric, lauric, myristic, palmitic and stearic acids have been prepared in good yield and some of their properties have been determined. The vinyl esters have been polymerized with benzoyl peroxide as catalyst and copolymerized with vinyl acetate or styrene. The simple polymers are soft and elastic, and the copolymers range in character from hard and glass-like to soft and elastic.

Vinyl esters of long-chain saturated fatty acids are a potentially valuable class of monomers since they are readily polymerized and copolymerized with other monomers. The properties of the products suggest many possible uses, such as pressure sensitive adhesives, water-resistant coatings, packaging materials, synthetic rubbers and many types of molded articles. In addition, the ability of the vinyl esters to copolymerize with commercially important monomers, such as vinyl acetate and styrene, suggests application as intramolecular modifiers. Products which are intramolecularly modified should not suffer from the disadvantages usually encountered with modifying agents which are mechanically admixed with a high-molecular weight polymer.

POTATO UTILIZATION INVESTIGATIONS

STUDIES ON DEVELOPMENT OF IMPROVED STORAGE METHODS FOR POTATOES

Considerable interest has been shown recently in development of better methods of storing potatoes, particularly in methods based on treatment with certain chemicals that retard sprouting and that would permit storage at temperatures higher than those in current use. Because of the late harvest and low temperatures prevailing during winter, Maine has good storage facilities where surplus potatoes can be kept until at least the following spring. By contrast, other Eastern states whose combined potato production greatly exceeds that of Maine have practically no storage facilities for potatoes. The refrigeration normally required for potatoes is expensive for milder climates.

Investigations carried out at some of the State Experiment Stations using carbon dioxide-enriched atmospheres for storage of potatoes have indicated that potatoes can be kept without deterioration using CO₂ enriched atmospheres. This suggested a possible method for storing and preserving the southern and intermediate potatoes. Accordingly a quantity of Maine potatoes were placed in storage at 68° F. Part of the potatoes were in an atmosphere containing 8% CO₂, and the other 12% CO₂. A control batch (without CO₂) was kept at the same temperature. After two months storage the control potatoes showed signs of sprouting. After the same period, however, the sprouting proceeded somewhat further in the potatoes kept at 8% CO₂, and was quite extensive in potatoes kept at 12% CO₂. The experiments were repeated using Florida potatoes at 68° F. but with only 3% CO₂. As in the previous experiments the tubers kept in CO₂ sprouted faster than the control tubers. From these results it appears that CO₂ promotes rather than retards the sprouting of potatoes, at least at the higher temperature ranges (68° F.).

There has been much interest in the use of the methyl ester of naphthalene acetic acid as a sprouting inhibitor, and in some localities the treatment has been carried out on a large scale. Storage tests on potatoes using this material were carried out at 68° F. in which confetti impregnated with the methyl ester was interspersed among the potatoes. On the whole the potatoes remained firm and there was little sprouting over the period of storage (November 1946-September 1947). In addition to observing the condition of the potatoes, chemical changes occurring during storage were determined, since there is little information regarding the chemical changes that occur during such storage. The results showed that during the first few months of storage there was only a slight change in the values for starch and sugar. After that there was a considerable decrease in the values for starch and a pronounced rise in the sugar content of the potato tubers, particularly in the values for non-reducing sugars. It is known that a similar change takes place in untreated potatoes, but this effect is attributed to sprouting, since the sprouts have a high sugar content. It is not clear at present why this phenomenon takes place in potatoes in the absence of sprouting.

Development of successful methods of storing potatoes at relatively high temperatures by treatment with compounds such as the methyl ester of naphthalene acetic acid offers considerable promise. Such methods would be of great importance for storage of surplus potatoes in the warmer regions of the United States. Before any conclusions can be drawn, however, a thorough study of the chemical changes taking place during storage under these conditions should be made.

STARCH ESTERS AND ETHERS

Commercial Development of Allyl Starch

Publication during the past year of improved methods for preparing allyl starch stimulated further industrial interest in this product. It has been reported that at least three concerns are building or operating pilot plants for the production of allyl starch. Two companies are now distributing samples to prospective customers. Another commercial concern making small quantities of allyl starch for its own evaluation has an undisclosed use for allyl starch which will require about 50,000 pounds annually. A company in Philadelphia found that addition of allyl starch improves the quality of printing ink, and they are ready to purchase allyl starch as soon as it appears on the market in commercial quantities. A number of other commercial organizations are investigating various applications of allyl starch.

CONVERSION OF SURPLUS POTATOES TO STABLE FORM

Dehydration for Feed Use

Because of the very large surpluses of potatoes during the past two or three years, and the perishable nature of the commodity, intensive studies have been carried out with a view to developing the most practicable and cheapest method of converting potatoes to stable form, to permit utilization in an orderly manner for livestock feed, or for industrial use. A survey of the various methods for drying potatoes in this country indicated that the most economic method was one based on washing the potatoes, grinding in a hammer-mill, dewatering in a continuous Davenport press, and drying in a direct heat Roto Louvre dryer. Because this process involves a serious fire and explosion hazard, pilot-plant experiments were carried out to develop an equally cheap but safe method.

The pressing operation mentioned above reduces the moisture content from 80 percent to about 65 percent, thereby greatly reducing the cost of drying. However, at 65 percent moisture the product sticks very badly unless the drying temperature is over 700° F. This requires a direct heat dryer, but this type is hazardous because of the tendency for the starch to form an explosive mixture with air. We found that by regrinding the pressed potatoes in a hammermill and mixing the product with dried potatoes sufficient to reduce the moisture content to 45 percent, sticking is overcome and drying can be done safely in a steam tube dryer.

This is the cheapest safe method of drying potatoes for feed which we have yet devised. The cost of the product, with potatoes at no cost, is estimated to be about \$23 per ton.

Conversion of Potatoes to a Crude Sirup

It has been suggested that good cattle feed ("potato molasses") can be prepared by hydrolyzing the whole potato and concentrating the hydrolyzate to improve its keeping qualities. Accordingly, experiments on hydrolysis of ground potatoes with hydrochloric and phosphoric acid were made, using concentrations of acid from 1 to 8 percent (based on the whole potato). It was found that the starch in whole potatoes hydrolyzes more slowly than pure starch and that hydrolysis with phosphoric acid is considerably slower than with hydrochloric acid. Efficient stirring reduces the time of hydrolysis. Since the mineral acid has to be neutralized after the hydrolysis, a noticeable salty taste is acquired by the sirup after concentration when two or more percent of hydrochloric acid is used. However, even the sirups prepared with the lower concentrations of acid are not sweet.

To improve the taste of the sirup the conversion of potatoes with malt diastase was studied, using 1, 3 and 5 percent of ground malt. Two hours were required to convert the potatoes with 5 percent malt, 6 hours with 3 percent, and 10 hours with 1 percent. On concentrating the converted liquid, a sirup of better taste than that prepared with acid was obtained. It was, however, not a sweet sirup.

In view of the fact that the product obtained by either acid or enzyme hydrolysis is not sweet, and in view of the high cost of producing such a product, it is not believed to offer any advantage over a straight dehydrated product for feed use.

TANNING MATERIALS, HIDES, SKINS AND LEATHER INVESTIGATIONS

DEVELOPMENT OF NEW AND POTENTIAL TANNING MATERIALS

Canaigre

Because of the importance of canaigre as a potential source of tannin, as well as a possible new field crop, a greatly intensified program is being developed on canaigre. Of first importance is the planting of sufficient acreage of the roots to assure more adequate supplies for experimental work and to develop more information on its cultivation. This is being carried out in cooperation with the Bureau of Plant Industry, Soils and Agricultural Engineering, and additional plantings are being made in the vicinity of Yuma, Arizona.

Field Studies

Experimental plots of canaigre, grown as a part of the cooperative program with the Bureau of Plant Industry, Soils and Agricultural Engineering at Lubbock, Texas and State College, New

Mexico, have been harvested and data recorded. The plots at State College, New Mexico, harvested after two years of growth, gave results of studies of: (1) various canaigre strains selected on a basis of good tannin content, root yields, and the production of roots well shaped for commercial handling; (2) water requirements; (3) effect of different spacing obtained by thick planting of canaigre seed followed by subsequent varying degrees of thinning.

Pilot-Plant Extraction Studies

A continuous process for the recovery of tannin extract from canaigre roots has been developed which should give about 78 percent yields. The process consists of adding sufficient water to dried, shredded roots to rehydrate them to about 30 percent moisture and allowing them to stand until they become limp and pliable. The roots are then passed through a comminuting machine and the cut roots are extracted with hot water in a countercurrent leaching unit. At regular intervals the material in each cell flows onto a vibrating screen and the oversize solids from the screen are pressed in a Davenport continuous press. The press liquor and the liquor through the screen are combined and returned to an adjacent cell toward the feed end of the extraction unit. The broken press cake is returned to its original cell. The head liquors, from the fresh feed cell, are clarified by means of centrifuges operated in series.

Fermentation of Canaigre Liquors

Bacterial fermentation of canaigre liquors by certain strains of Aerobacter results in almost complete destruction of the sugar. Tanning extracts prepared from such fermented liquors are quite satisfactory, being high in tannin and having high purities. Since the successful commercial development of canaigre as a source of tannin may depend in part on the economic utilization of its starch and sugar, it is important that the products obtained by the fermentation of these materials be determined. Members of the Aerobacter group usually produce chiefly carbon dioxide, hydrogen, 2,3-butanediol, acetoin and ethanol. This work is underway.

Sumac

Drying Studies: Since it has been previously shown in these studies that several domestic sumacs are equal or superior to that imported from Sicily, additional studies were undertaken to determine the effect of drying methods on leather quality. During the summer of 1946 large quantities of leaves and stems of Rhus glabra and R. typhina were collected and dried by various procedures. Nine lots of each species of sumac were obtained. These were used in tanning about 200 sheepskin skivers. The tanning and finishing of the leather have now been completed and a preliminary grading of the skins has been made. The results indicate that the method of drying does have some influence on leather quality. However, the final data are not yet complete.

Tanning Studies: A further comparison of physical properties of sheepskins tanned with Sicilian and three domestic sumacs shows

no marked differences in properties that can be attributed to species of sumac used. These results give further proof that domestic sumacs can be successfully used as replacements for Sicilian sumac.

Scrub Oak

Analytical Studies: Further analytical studies of the tannin content of scrub oak bark have been carried out. Since the proposed development of scrub oak bark contemplates the use of trees of very small diameter and also the limbs and branches as a source of bark, it is important that information be available concerning the tannin content of bark from small trees and branches, and its relation to the tannin content of bark from trees of larger diameter. The average tannin contents of the barks from the various tree parts were not essentially different, indicating that bark taken from trees of small diameter and from tops of trunks and from limbs should be equally as satisfactory, as regards tannin content, as bark taken only from tree trunks.

DEVELOPMENT OF COMBINATION VEGETABLE-ALUM AND OTHER TANNAGES

Alum Retannage

In connection with the leather research program of the Office of the Quartermaster General, collaborative work has been continued on the development of alum retannage of vegetable tanned leather to produce more serviceable insole leathers. By use of alum liquor "masked" by sodium acetate, leathers have been made that contained more than three percent Al_2O_3 and showed no shrinkage when boiled in water for more than three minutes.

Tests have also demonstrated that the retannage can be accomplished satisfactorily in "rocker" vats. The gentle motion of the leather in the "rockers" proved to be as effective in fixing the required amount of Al_2O_3 as was the more violent motion in tanning drums. The "rocker" tannage has the advantages of (1) using easily available vats instead of tying up more costly tanning drums; (2) saving in power costs; and (3) eliminating the risk of grain damage resulting from the action of the drums.

In the alum retanning process it has been found necessary, in order to obtain leather of the required Al_2O_3 content, to use in the tanning liquors more than twice the amount of Al_2O_3 that is finally fixed in the leather. It would be advantageous therefore to salvage, adjust, and reuse the spent liquors. A successful system was developed which utilizes about 85% of the aluminum added.

CHEMICAL AND PHYSICAL PROPERTIES OF COLLAGEN

Electron Microscopic Studies of Collagen

Through electron microscopy a beginning has been made in determining the structure of collagen fibrils, which are the principal sub-microscopic constituents of skins, and the manner in which

fibrils are altered in the preparation, testing, and use of leather.

Owing to the inadequacy of present sectioning techniques, it is necessary to prepare specimens for electron microscopic examination from mechanically disintegrated collagenous tissue. Disintegration by grinding, teasing, scraping, or the complex action of a Waring blender has been effective. An aqueous suspending medium seems essential for complete dispersion. Specimens obtained by evaporating water from dispersed tissue usually have collagen fibrils as their most prominent component, but structural details are masked by accompanying smaller, formless particles not yet characterized. Separation of this debris from the collagen is accomplished by repeated differential centrifuging. Observations have been limited largely to the collagen because it is the principal protein of skin, it has a macromolecular structure discernible by electron optics and reliable correlative data are supplied by X-ray diffraction and by chemical analysis.

Electron microscopic studies on shrinkage of collagen have been carried out. From present evidence, the shrinkage of collagen may be described as follows: Initially the units are orderly arrays of linearly extended collagen molecules, the striated fibrils. When plasticized by heat and water, the interactions maintaining the orderly structure are weakened and the natural contractile tendency of long, flexible molecules becomes predominant, shortening the fibril as a whole.

The shrinkage temperature is a useful index of the resistance of collagen to swelling by water or a water solution. In general, the shrinkage temperature is raised by tanning and lowered by peptizing electrolytes and by protein degradation.

The effect of sodium fluosilicate on collagen fibrils was determined because of its use as a preservative in the salt curing of hides. On treatment with 1 percent fluosilicate, the fibrils were considerably swollen and frayed, as when treated with dilute acid. Substituting 10 percent sodium chloride and 1 percent sodium fluosilicate for the fluosilicate alone, the fibrils were normal and the shrinkage temperature (measured in the solution) was 67°, even though the pH was 3.6. Evidently the swelling effect of low pH was just offset by the deswelling effect of the sodium chloride. The fluosilicate ion itself apparently has no deleterious effect on the collagen fibrils.

RESEARCH AND MARKETING ACT PROJECTS

Projects assigned to the Eastern Regional Research Laboratory under Title I, Section 10 (a) of this appropriation include the following:

- RMA-113. Disposal of dairy wastes.
- RMA-125. New and improved uses of deciduous fruits, especially to prevent waste.
- RMA-129. Investigations of tanning and development of new tanning materials.
- RMA-132. Processing Nicotiana rustica as a source of nicotine.
- RMA-166. Conversion of potatoes to more stable forms and products; and new methods of using potatoes for feeds and non-food uses.
- RMA-200. Utilization of wool grease and other wool byproducts.

Since funds were not available until the last two months of 1947, most of the effort so far (January) has been concerned with recruitment of personnel, literature surveys, purchase of necessary special equipment, conferences with cooperating groups, and other steps necessary for the initiation of experimental work. Although the professional recruitment problem is difficult, considerable progress is being made, and it is anticipated that within the next few months the additional staffing will be completed. The projects on potatoes, tobacco and tanning materials involve pilot plant scale studies at the outset. Rapid progress is being made in the design of these experimental engineering units and in the procurement of necessary equipment.

NORTHERN REGIONAL RESEARCH LABORATORY

G. E. Hilbert, Director

AGRICULTURAL RESIDUES UTILIZATION INVESTIGATIONS

COMPOSITION AS AFFECTED BY RAW MATERIAL PROCESSING AND STORAGE

The whole program of industrial utilization of agricultural residues depends first on low cost, simple and efficient methods of collecting and storing them. Real progress has been made in the collection and storage of wheat straw, as the result of two meetings held at the Laboratory in 1947 between members of the farm equipment and strawboard industries, together with the Fibrous Agricultural Residues Committee of TAPPI and the Crop Residues Committee of A.S.A.E. Many of the one-man pick-up balers as generally operated do not produce a sufficiently dense bale to withstand transportation and storage. When properly operated some of these machines can produce a bale of 9.5 lbs/cu. ft., which is desired. An educational program is underway by the two industries to teach operators how to bale straw correctly. In addition, a number of the equipment companies have undertaken to improve their machines for baling straw. By bringing the industrial men together a mutual interest and understanding of this important problem has resulted.

A cooperative project to develop chemical treatment of the top bales of straw ricks to reduce decomposition of straw in mill yards is under way by seven strawboard and seven chemical companies, the Fibrous Agricultural Residues Committee of TAPPI, and the Laboratory. Preliminary results are promising. Results will be correlated by the Laboratory after the bales have been in storage for one year.

A moisture meter which accurately measures the moisture in baled straw has been developed cooperatively by the Laboratory, the TAPPI committee, and an instrument manufacturer. This instrument should be satisfactory also for determining the moisture content of baled hay.

ALCOHOL AND OTHER LIQUID FUELS

Synthetic Liquid Fuels Investigations

The research work under the Bureau's Synthetic Liquid Fuels Project is closely coordinated with that of the Northern Laboratory. The process for hydrolyzing agricultural residues to sugars for conversion into liquid fuels basically consists of converting one of the fractions of agricultural residues, pentosans, to pentose sugars and, subsequently, converting the cellulose fraction to dextrose. The pentose sugars may be fermented to the liquid fuels butanol, acetone, isopropanol, and ethanol, or they may be converted to furfural. The dextrose, of course, may be fermented to the liquid fuel ethanol.

A semi-works plant for studying this process has been in operation for about one year. Approximately 400,000 pounds of cobs have been processed. One hundred runs of 4,000 pounds of cobs each have indicated that pentosans in corncobs may be hydrolyzed in 93-percent yields to conversion products, 87 percent of the pentosans being converted to pentose sugars and 6 percent to furfural. Since cobs contain about 32-percent pentosans, a little more than 600 pounds of pentose sugars may be produced from one ton of dry cobs. These methods of producing pentose sugars are presently being used by two industrial concerns. One concern is producing crystalline pentose sugars on a small scale, and the other is investigating the production of both pentose sugars and dextrose from corncobs. The second step in the semi-works plant for converting cellulose to dextrose is now under study.

The Butanol Fermentation of Pentosan Hydrolysates

In the fermentation of pentosan hydrolysis liquors to butanol, acetone, and ethanol, considerable simplification of the fermentation mash and procedure has been made by the Laboratory. Control of the initial oxidation-reduction potential of the medium to insure anaerobic conditions has proved necessary for satisfactory fermentations. Copper, resulting from mild corrosion of the hydrolysis equipment, and at least one other unidentified toxic factor, are present in the pentosan hydrolysates and deleteriously affect the multiplication of organisms. Substances beneficial and essential to the fermentation are also present. Removal of the toxic principles alone from the liquors prior to fermentation has been found possible by adequate pretreatment of the hydrolysis liquors with iron powder. By eliminating unnecessary chemicals, the cost of chemicals required to supplement the pentosan hydrolysate liquors has been reduced from 2.43 cents to 0.35 cents per pound of mixed solvents produced. An economical and practical procedure to remove only the deleterious substances is now being developed.

An industrial butanol fermentation company has evidenced considerable interest in the process and will shortly test it in their plant.

Motor Testing of Synthetic Liquid Fuels

(See "Alcohol and Other Liquid Fuels" under the report "Corn, Wheat, and Other Cereal Crops Utilization Investigations.")

PLASTICS, ELASTOMERS, AND RESINS

A study of methods and costs of grinding nut hulls and nut shells, corncobs, and certain other residues for use as fillers in plastics, as soft-grit blasting materials, abrasive and polishing materials is well under way. Many of the nut hull and nut shell flours appear suitable as fillers in phenol formaldehyde plastics. The flours also appear useful as extenders in plywood adhesives, lowering the cost of such adhesives 25 to 30 percent. Lignin

obtained from the process of saccharifying corncobs makes a good extender for such adhesives. Conditions have also been found for its use in making phenolic-aldehyde plastics.

SYNTHETIC ORGANIC CHEMICALS AND INTERMEDIATES

Chemicals from Furfural

A number of rubber and plastic manufacturing companies have reported that 1-5 pentanediol, for which the Laboratory developed a synthesis, has good qualities for preparing linear polyesters of high molecular weight as well as for plasticizing vinyl polymers. Another compound, delta-valerolactone, synthesized by the Laboratory is reported by a large manufacturer of resins to show promise of industrial use.

A prominent drug manufacturer has applied for license under the Laboratory's U. S. Patent 2,424,184 for the manufacture of Noval alcohol from furfural. This alcohol is an important intermediate in the manufacture of anti-malarial drugs.

Work has been completed on the synthesis of various derivatives of 4,4'-dichlorodibutyl ether. They may be useful as plasticizers. Samples of several have been distributed to interested companies. By stepwise hydrogenation of furfural-acetonefurene, which may be obtained in 75 percent yield from furfural, three new compounds have been obtained: furyl-beta-furylethyl ketone, 1,3-difuryl-propanol-1, and 1,3-ditetrahydro furylpropanol-1. The last compound is of interest because like "F-alcohol" (used by the Germans), it has a high boiling point, and is not only completely soluble in water but also soluble in many organic liquids.

Considerable attention has been devoted to developing a process for crystallizing xylose from the pentosan hydrolysate of corncobs. A large number of samples of xylose have been distributed. Several individuals and agencies are interested in studying the pharmacological and physiological action of xylose. A small company in Colorado is manufacturing good crystalline xylose from cobs.

STRUCTURE AND OTHER BASIC INVESTIGATIONS

The Structure and Synthesis of Gymnosperm Lignin

For 70 years chemists have sought to determine the structure of lignin. Dr. Alfred Russell, an authority on the chemistry of tannins, joined the Laboratory staff early in 1947 to undertake the lignin problem. From a consideration of the known degradation products of lignin, Dr. Russell in a few months reached the conclusion by brilliant inductive reasoning that gymnosperm lignin is a poly-8-methoxydihydrobenzopyrene, a relatively simple

structure; closely related to that of the tannins, flavones, flavonones, and flavanols, important plant pigments. Following this, gymnosperm lignin was synthesized from vanillin monoacetate by the Fries rearrangement. Quantitative comparison has been made by analysis and analysis of derivatives, together with X-ray and absorption spectra. The natural and synthetic lignin seem inescapably identical. Analogous lignin-like products have been produced by the same methods starting with ortho and with para hydroxy benzaldehyde.

CELLULOSE PULPS FOR PAPER AND BOARD MANUFACTURE

The strawboard industry is awake to the necessity of improving the quality of 9-point corrugating board. They are supporting the Laboratory's work and are looking generally to it for help. Publications have been issued on the new process for cooking strawboard pulp by the use of neutral sodium sulfite and lime. A large number of the mills are working with this process in their operations.

Studies on the comparison of the physical properties of straw pulps for fine papers and a variety of commercial wood pulps show that, with the exception of tearing strength, straw pulp is stronger than hardwood soda and equal to softwood sulfite pulps. A study of the various methods of pulping wood as applied to wheat straw shows that only alkaline cooking chemicals are suitable for straw. The Laboratory's new process for producing fine paper pulps from wheat straw in screened, bleached yields of 50 percent based on dry straw has been reported. By comparison, yields of about 40 percent had been obtained heretofore. A number of paper mills are becoming genuinely interested in the process. The shortage and increasing cost of pulpwood is causing a changed viewpoint in the paper industry.

BUILDING AND CONSTRUCTION MATERIALS

The first two papers on the fundamental relationship of fibrous agricultural residues to the manufacture of insulating building board are in course of publication. These will be followed by a publication on the process for manufacturing exceptionally high quality insulating board from wheat straw, in which process there is some industrial interest. The Laboratory's fundamental work has shown, surprisingly, that wheat straw is superior to cornstalks and probably to bagasse for making building materials. Four hundred 3' x 3' box boards are being made from wheat straw for completing, with a manufacturer of wirebound boxes, a basic study on the use of a special type of board for such use. The study involves a great deal of testing work in the industrial box-testing laboratory.

CORN, WHEAT, AND OTHER CEREAL CROPS UTILIZATION INVESTIGATIONS

COMPOSITION AS AFFECTED BY VARIETY, BREEDING, ENVIRONMENT, AND MATURITY

Oil Content of Corn as Influenced by Heredity

Additional analyses of both single crosses and pure inbred lines have confirmed the influence of heredity on the oil content of corn and have furnished information on the classification of present available inbred lines with respect to their ability to increase the oil content when used in crosses. In close collaboration with Experiment Station agronomists, about 600 samples of corn have been analyzed to furnish these agronomists with information on their progress in breeding for high oil content. About 400 of these samples came from Indiana; 340 from Illinois, and the remainder from Ohio.

Comparison of Corn Starches at Various Stages of Kernel Maturity

Starch in the grain of 3 corn varieties, Iowa 939 hybrid dent, Iowax 1 hybrid waxy dent, and Golden Cross Bantam sweet was studied at 5 stages from 12 days after pollination to full maturity. Over this period the starch increased from 10 to 65 percent of the grain (dry basis) in the waxy and non-waxy dent and from 5 to 26 percent in the sweet corn. Average starch granule diameters increased from 2.7 to 9.9, 3.0 to 9.1, and 1.3 to 3.5 micron in the dent, waxy, and sweet varieties, respectively. Water binding capacity of the starch decreased 50 percent by maturity. The amylose content of the non-waxy starch at maturity was about double the early values. The most rapid changes in starch properties occurred during the first 35 days after pollination.

ALCOHOL AND OTHER LIQUID FUELS

Single-Cylinder Engine Tests on Motor Fuels

Preliminary studies on the effect of supercharging on absolute ethyl alcohol have been completed in a single-cylinder test engine. Under these conditions alcohol is equivalent to a high octane fuel. Since many recently discovered domestic and foreign petroleum fields produce high sulfur crudes, the problem of sulfur in gasoline is becoming acute. The presence of sulfur lowers the effectiveness of tetraethyl lead in raising the octane number, and differences in octane numbers between the same base gasoline with and without sulfur may amount to four or more units depending on the sulfur compounds encountered. Studies with alcohol blends so far have indicated that the type of sulfur compound as well as the amount are important, and it appears that in some cases ethanol may partially "neutralize" the detrimental effect of sulfur while in others no significant difference was observed. The liquid-vapor equilibrium of ethanol-isooctane systems has been measured with high precision at 25° and 50° C.

Multi-Cylinder Engine Tests on Motor Fuels

Performance of a Chevrolet engine using blends containing from 5 to 20 percent alcohol showed that little or no change was needed

in the engine to efficiently use the 5 percent blend, and 10 and 15 percent blends could be used satisfactorily by changing carburetor jets to the standard commercial rich jets. High compression (9:1), high-speed engines are being designed for future automotive use. Such an engine requires high octane fuel (90 or over), but a 20 percent alcohol blend with regular gasoline gives satisfactory performance. Preliminary studies have shown great promise in the use of alcohol-water by injection and these studies are being actively continued under a Research and Marketing Act project.

Fungal Amylase by Submerged Culture

The yield of alcohol from the fermentation of corn mashes saccharified with mold culture filtrates appears to correlate more closely with the maltase than with the α -amylase activities of fungal enzyme preparations. By slightly modifying the media formerly used, the maltase activities were increased. The use of such high potency fungal enzyme preparations permitted a substantial reduction in the amount required to saccharify the corn mash and still give the same yield of alcohol on subsequent fermentation with yeast. When equal quantities of enzyme preparations were used, higher yields of alcohol resulted from the use of the preparation higher in maltase.

Several distilleries are now investigating the use of submerged fungal enzymes as saccharifying agents. A report from one of them on the use of *Aspergillus niger* NRRL-337 appeared in Ind. Eng. Chem. (1947) 39, 1315. Increased yields of alcohol from the fermentation of corn mashes saccharified with fungal enzymes over malt controls were consistently obtained, thus confirming our Laboratory findings.

FIBERS, FILMS, PROTECTIVE COATINGS, AND ADHESIVES

Zein Fiber

The Laboratory's work on the continuous wet spinning of fibers from zein has stimulated the interest of several commercial companies. One company has initiated intensive work on this fiber. As of January 1948, this company is manufacturing approximately 100 pounds of zein fiber daily, and plans to increase this amount by many times. The fiber made by this company appears to be useful for blending with wool, rayon, cellulose acetate, nylon, and cotton fibers.

Improvements in the preparation of zein fiber have been made by the Laboratory. Of greatest importance was the discovery of a new method for treatment which permits omission of the acetylating treatment and which gives a fiber having excellent resistance to wool dyeing conditions. Results show a fiber with a strength of 1.4 grams per denier, dry, and 0.7 grams per denier, wet, and a shrinkage of 10 percent or less after the severe conditions of acid-dyeing.

SYNTHETIC ORGANIC CHEMICALS AND INTERMEDIATES

Acetal Derivatives of Dextrose

Following the studies on the formaldehyde derivatives of dextrose, attention has been given to the preparation and properties of the higher acetals of dextrose and gluconic acid. Monobutylidene glucose, dibutylidene glucose butyral, tributylidene, and tri-isobutylidene sorbitol, and the triketals of gluconic acid, sorbitol, and glucose with cyclohexanone have been synthesized. Most of the derivatives are colorless, viscous liquids or glassy substances having low vapor pressures and good heat stability. They are soluble in the common organic solvents and insoluble in water. The products are being investigated for use as plasticizers.

Dextrose Condensation Products

A new method has been discovered for the preparation of alkyl glucosides from alcohols and dextrose, employing boron trifluoride as a catalyst. Crystalline α -methyl glucoside in 60 percent yield was obtained from the mixture of the α - and β -isomers. The methyl glucosides of the disaccharides, maltose and lactose, have also been prepared directly by this method. An efficient means for separating the isomeric disaccharide glucosides would be particularly valuable because of the difficulty in preparing the α -glucosides.

Starch Esters

Aromatic isocyanates which have recently become available industrially have been found to react readily with starch and its fractions without pretreatment of the starch. The tricarbanilates are levorotatory in pyridine solution, and a significant difference in rotation is shown between the linear and non-linear starch fractions. Starch tricarbanilate is the first ester found that can be separated into linear and non-linear fractions by organic solvents. Partial esterification with difunctional isocyanates can be controlled to give starch products either totally or partially resistant to boiling water. Uses for these products are being sought as water-resistant dusting powders, fillers, and stable organic carriers.

NEW AND IMPROVED FEED OR FOOD PRODUCTS

Production of Riboflavin

Work has been continued on the production of riboflavin (vitamin B₂ or G) by fermentation with the Ascomycete *Asibya gossypii*, with promising results. In the pilot plant, studies have been made to determine the optimum conditions for sterilization of the medium to be fermented. Experiments are now being conducted to determine the optimum rate of aeration and the optimum composition of the fermentation medium.

This aerobic fermentation is conducted in an inexpensive medium of 2 percent glucose, 0.75 percent each of corn steep water solids

and animal stick liquor (a byproduct of wet rendering). The medium, adjusted to pH 6.5, is sterilized, cooled and inoculated with a day-old liquid culture of Ashbya gossypii. Sterile air is supplied at a rate of 1/4 to 1/2 volumes per volume of medium per minute. Fermentations are complete in 72 hours and yields of 400 micrograms of riboflavin per ml. of fermented liquor have been obtained frequently in 300-gallon batches. The highest yield obtained so far has been 520 micrograms. Because the conditions of operation have been varied the results have varied considerably.

This fermentation process has attracted wide industrial interest. Several companies are conducting pilot plant investigations, and one other has engaged chemical engineering counsel for plant design and erection.

EXTRACTION, RECOVERY, AND PROPERTIES OF SPECIFIC CARBOHYDRATES, OILS AND PROTEINS

Wet Milling of Damaged Corn

The amount of free sulfur dioxide in the steepwater on samples of sound and damaged corn was determined at intervals throughout the steeping process. In confirmation of our earlier findings on the beneficial effect of increased sulfur dioxide in the steeping of damaged corn, it was found that damaged corn took up more sulfur dioxide than sound corn. Further work on the reaction between damaged corn constituents and sulfur dioxide is required to establish the best conditions for processing such corn.

Wet Milling of Grain Sorghums

Because of the development of varieties that can be harvested with the combine, grain sorghum has become an important crop in the Southwest, and the Corn Products Refining Company is building a plant at Corpus Christi, Texas, capable of wet-milling 20,000 bushels of sorghum per day. The company has requested the aid and cooperation of this Laboratory in solving some of the problems encountered in the processing of grain sorghum.

When the wet-milling of waxy grain sorghum was being studied at this Laboratory four years ago for the production of starch during the war, it was found that the color-bearing layer and hull of the kernel must be removed to insure the production of "white" starch. The cleaning was accomplished by passing the grain through a barley pearling machine. The resulting "dehulled" or "pearled" grain fraction could be wet-milled with ease and the quality of the products extracted from it was improved.

Grain sorghum, typical of that grown in the Southwest, was obtained for test purposes. Part of the grain was set aside to be processed without preliminary treatment; the remainder was pearled to remove the hulls. In this latter operation about 83 percent of the grain was recovered as dehulled sorghum and 17 percent was removed as hulls. The waxy material was extracted

from the hulls and it was determined that approximately 1/3 pound of purified wax can be obtained from the hulls which are removed from each 100 pounds of grain.

Tests have been conducted on a laboratory scale on the wet-milling of the whole grain as compared with the processing of the dehulled or pearled sorghum. All tests were performed under identical operating conditions. It was found that the pearled grain is more easily processed and yields much more starch of better quality. Furthermore, the waxy material, similar to carnauba wax, can be extracted from the hulls that are removed during the pearling operation.

Whole grain was wet-milled in a series of experiments in which the concentration of sulfur dioxide in the steepwater at the beginning of the steeping period was varied from 0.10 to 0.25 grams per 100 cc. Only minor differences in processing were noticeable. Since a concentration of sulfur dioxide as high as 0.25 grams per 100 cc. of steepwater did not materially damage the starch, a series of runs was conducted in which this concentration of sulfur dioxide was used in all tests and the steeping temperature was varied from 110° F. to 150° F. A marked decrease in starch recovery and viscosity resulted from an increase in steeping temperatures. Although optimum steeping conditions for wet milling grain sorghums undoubtedly vary slightly with the variety of grain being processed, data have been obtained which indicate that the concentration of sulfur dioxide in the steepwater should be approximately 0.20 to 0.25 grams per 100 cc., and the grain should be steeped for a period of 48 to 72 hours at a temperature of 110° F. to 118° F.

Production of Undevitalized Wheat Gluten

In an effort to find an improved commercial method for removing water from doughy wheat gluten so that a dry undenatured product could be produced more economically, several procedures have been investigated. The most promising of these include (a) the extrusion of a thin film of wet gluten into a drying tunnel, (b) the extraction of water from wet gluten with acetone, and (c) the drum drying of an ethanol dispersion of wet gluten.

The dried products obtained by using these procedures were compared with costly present-day commercial gum gluten which is made by drying small pieces of wet gluten in vacuum tray driers. In this comparison two tests were conducted. One was based on the capability of the dried product to be reconstituted into a dough when rewet with water. The other, a chemical test, was based on the amount of protein in the dried gluten which could be solubilized in acetic acid. As measured by the above tests, all three methods gave undenatured gluten.

Since the largest use of undenatured gluten would probably occur in the fortification of low-protein flours, it was deemed advisable to submit the glutens to practical baking tests. Through the courtesy of the Agricultural Experiment Station at the North Dakota Agricultural College, blends of starch and dried glutens as well as

blends of low-protein flour and dried glutens were subjected to baking tests in their laboratories. Also samples of commercial gum glutens and a standard gluten prepared in the laboratory by officials of the experiment station were tested.

The baking tests revealed that commercial gum glutens and those glutens produced by our drying procedures are unsuitable for the preparation of synthetic baking mixes which are comprised of blends of wheat starch and dried undenatured wheat gluten. The fortification of low-protein flours with gum gluten appears impractical, since no appreciable increase in dough strength or bread loaf volume can be detected when the protein content of flour is increased from 11 to 13.5 percent even by the addition of undenatured gluten of the highest quality (North Dakota Experiment Station standard gluten).

Comparative Study of Starches from Various Seeds and Stems

The search for starches of unusual type and properties has been continued. Starch was separated from 12 species of plants representing 9 botanical families. Starch of horsechestnut, oak, buckwheat, rice, sorghum, mung bean, field pea, canna, iris, dasheen, yautia, and sago were included. Gel characteristics and paste clarity could not be correlated with the source of the starch. The amylose content ranged from about 11 percent in yautia starch to 35 percent in field pea starch. No strikingly unusual starches were found, and the differences noted indicated that the factors of botanical source and site of deposition were not the most important influences on the character of the starches.

Water Absorption Studies on Starch and Its Constituents

An apparatus for measuring the surface area of solid starch granules (B.E.T. method) has been constructed and used on a single sample of dry corn starch. The surface area for nitrogen gas sorption was found to be surprisingly low, 0.83 square meter per gram. This is about one-half the area reported for paper or cotton fibers. Several methods of measuring the specific gravity of starch at various degrees of hydration were investigated but none was found sufficiently accurate to be satisfactory. Microscopic measurements of linear dimensions of single granules (one sample) exposed to atmospheres of controlled but variable humidity were accurate and showed an increase of 9 percent in linear dimension in going from a dry to a water-saturated atmosphere. Between 7 and 24 percent relative humidity atmospheres, starch increases only 1.05 percent in volume compared with 1.60 percent calculated. The difference represents water entering existing pore volumes of the granule. These pore volumes, however are not available to nitrogen gas as shown by adsorption isotherms so that there is evidence for a skin around the granule which is impervious to nitrogen, but permeable to water vapor.

Starch Hydrolysis--The Action of Cerecal Amylases on the Amylose-Fatty Acid Complex

The incomplete conversion of starch to sugar and the incomplete solubilization of starch sizes are factors of practical significance in the fermentation and textile industries. The fat in commercial corn starch is sufficient to separate about 5 percent of the weight of the starch as the amylose-fatty acid complex. Initial results have shown that under conditions that would give complete starch solubilization the fatty acid complex is but half destroyed.

Degradation of Amylose by α -Amylase

Conditions and methods have been developed for the controlled degradation of amylose in 4 percent solutions by pure α -amylase without interference by amylose retrogradation. The method has been found applicable to 8 percent suspensions of corn amylopectin. The method is being applied to the investigation of the specific action of this enzyme on amylose and amylopectin as related to the industrial utilization of starch and the preparation of polysaccharides of definite molecular size.

CHEMICALS DERIVED FROM FERMENTATION

Polymyxin

The first report of the isolation of the new antibiotic "polymyxin" from Bacillus polymyxa issued from this Laboratory in April 1947. Since that time, publications from the American Cyanamid Company and the Wellcome Physiological Research Laboratories in England describe independent discovery of the same antibiotic. Joint efforts have so far revealed that polymyxin (1) can be prepared in quantity and in form for clinical use, (2) is a basic polypeptide, (3) is relatively non-toxic, and (4) is highly active in vitro against the gram-negative organisms causing typhoid, plague, one type of pneumonia, brucellosis, tularemia, whoopingcough, etc.

In this Laboratory a superior plate assay method has been designed for the estimation of polymyxin using Brucella bronchiseptica as the test organism. Studies on the composition of the culture liquor have shown that the expensive yeast extract originally used can be replaced by much cheaper protein sources, with considerable increase in yield in some cases. Modification of strains of Bacillus polymyxa has yielded new forms that produce five times as much polymyxin as the best original strain. Polymyxin hydrochloride has been prepared in sufficient quantity for tests at the National Institute of Health on experimental animals infected with undulant fever organisms. Over a dozen inquiries concerning polymyxin have been received from various pharmaceutical houses and universities, and sub-cultures of the B. polymyxa organism and polymyxin hydrochloride standard have been supplied to these interested parties.

Utilization of Bionic Acids

The large-scale fermentation of lactose and maltose by Pseudomonas graveolens to give lactobionic and maltobionic acids is essentially similar to the small-scale (1947 report). One company is now conducting feeding experiments and investigating the generation of calcium lactobionate in the manufacture of processed cheeses. If these stand long on store shelves, the lactose of the incorporated whey sometimes crystallizes, and customers complain of "glass" in their cheese. The fermentation of the lactose to the very water-soluble calcium lactobionate may eliminate this problem. The convenient preparation of these acids on a large scale is intriguing, and together with their salts they may be expected to find some application in pharmaceuticals as well as in the food industry.

Other Chemicals and Developments

The production of citric acid in submerged culture and the fermentation resulting in α -ketoglutaric acid are being studied intensively. In the last two years an active part has been taken in a collaborative malt program designed to elucidate the important enzyme components in malt and to study their measurement. Extending the work mentioned in last year's report regarding the vitamin content of grain sorghum, it has been found, with the help of several Experiment Stations, that the niacin content of corn is also genetically controlled and, to a certain degree, may be altered by planned hybridization.

SOYBEANS AND OTHER OILSEED CROPS UTILIZATION INVESTIGATIONS

COMPOSITION AS AFFECTED BY VARIETY, BREEDING, ENVIRONMENT, AND MATURITY

Composition of Various Oilseeds

In cooperation with the Illinois Agricultural Experiment Station, 123 oilseed samples were analyzed. These comprise 91 sunflowers, 26 castor beans, 4 perilla, and 1 each sesame and rape. It was found that ground castor beans quickly developed free fatty acids which interfered with the subsequent analysis of the oil so that immediate oil extraction was necessary after grinding the castor bean seed. Castor bean acreage is rapidly increasing in Illinois, and sunflower acreage is being shifted towards the south to obtain more favorable growing conditions.

Analysis of Oils

Further work on the composition of oils using the thiocyanogen and alkali conjugation methods of analysis has been carried out in collaboration with groups from the American Oil Chemists' Society. The alkali-conjugation of oils followed by spectrophotometric examination has given good results in collaborative tests but requires additional calibration for high accuracy. The thiocyanogen method, in spite of much investigation, is still not satisfactory for routine use because of the lack of reproducibility. Evidence has been obtained that variations in the reagent preparation are responsible for the erratic results frequently obtained.

PLASTICS, ELASTOMERS, AND RESINS

Norelac

The development of a process to produce synthetic resin, Norelac, by the chemical modification of soybean oil has been previously reported. Evaluation of Norelac has progressed through the pilot plant and semi-commercial stages. On July 1, 1947, an industrial company began full-scale production in a new plant having a capacity of 100,000 pounds of finished resins per month. Norelac is used primarily as a heat-sealing agent for sealing various packaging materials.

FIBERS, FILMS, PROTECTIVE COATINGS, AND ADHESIVES

New Vegetable Adhesives

This Laboratory has been studying the preparation of an unusual vegetable material obtained from an aqueous extract of soybean meal. It was first isolated from alcoholic extracted flakes. This gel is suitable for whips and meringues, but it also possesses some of the characteristics of egg albumen. It gives a heat irreversible gel and possesses uncommon adhesive characteristics. Since its adhesive characteristics are unusual, an attempt was made to prepare a similar product from hexane-extracted flakes. Commercial hexane flakes were extracted with alcohol, the resultant product extracted with water, and the extract spray-dried to give the adhesive product. Although its flavor is not as good as the aqueous extract of alcohol-extracted flakes, its adhesive characteristics are almost identical. A process for pilot plant work has been devised to explore the manufacture of this adhesive. A cooperative agreement with an interested industrial company has been made to put the process into operation. The adhesive appears to be adapted to a wide variety of uses including sealing of cork to metal, paper to metal, remoistening and heat-sealing adhesives, as well as certain food uses where its flavor is partly masked.

Soybean Adhesive for Shotgun Shell Casings

The development by the Laboratory of a soybean protein adhesive for use in the manufacture of shotgun shell casings was reported last year. In cooperation with the Laboratory a large manufacturer of cartridges conducted full-scale plant trials to evaluate this adhesive and a large number of experimental shells were made for testing. Aging tests and shooting trials showed that these shells were equal or superior to those that the company had manufactured previously. Actual commercial use by this company of soybean protein adhesive was initiated in May 1947, and it is estimated that this adhesive has been used in about half of all the shotgun shells made since last summer.

Conjugation of Oils by Chemical Treatment

Last year a new process for the isomerization of vegetable oils was reported briefly. This process is the treatment of the oil with

tertiarybutyl hypochlorite (TOCl), and subsequent dehydrohalogenation of the treated oil. TOCl introduces approximately 1 equivalent of chlorine into the fat acid chain per mole of TOCl: Six tenths equivalent of this chlorine is very active and easily removed by treatment with steam at 150° C., 0.2 equivalent is less active but can be removed by somewhat more vigorous treatment, and the final 0.2 equivalent is very inert and difficult to remove. Encouraging reports have been received on the utilization of this material for linoleum, varnishes, and paint oil extenders.

NEW AND IMPROVED FEED OR FOOD PRODUCTS

Flavor Stability of Soybean Oil

Research on suitable additives for deodorization of soybean oil has been extended to include new materials such as tartaric, tricarbyllic, and other polyfunctional acids, and sorbitol and other alcohols. One function of these compounds is the inactivation of trace metals as catalysts for oxidation. The addition of 0.3 to 3 parts per million of iron as ferric chloride or ferric stearate increases the rate of autoxidation of fats and this effect can be partially to completely stopped by the addition of 0.001 to 0.1 percent of citric, tartaric, and other acids, and sorbitol and other polyhydric alcohols. Commercial tests with citric acid have been conducted by formal cooperation with Armour and Company and through informal cooperation with another company. Sorbitol has been tested on a commercial scale with one company. According to the analysis conducted with the Laboratory's taste panel, the flavor of the treated oils prior to aging was equal to or better than that of the untreated oils; they were also more stable to both accelerated and room-temperature aging. The companies cooperating on the use of citric acid checked the Laboratory's analysis with their own taste panel.

The techniques of organoleptic evaluation are being studied and evaluated. The Laboratory's present techniques are not completely satisfactory but the placement of the collection of organoleptic data from oils on a sound scientific and statistical basis has been achieved.

EXTRACTION, RECOVERY, AND PROPERTIES OF SPECIFIC CARBOHYDRATES, OILS, AND PROTEINS

Fractionation of Soybean Oil by Liquid-Liquid Extraction

A number of tests were conducted to study the effect upon the degree of separation obtained when the temperature of the water in the jacket of the extraction column was varied. The temperature range used was from 90° to 140° F., the reflux rate was adjusted to give a 50-50 ratio of products, and the furfural entering the column was cooled to below room temperature and saturated with naphtha. Twenty parts of furfural to one of oil was used for these tests. Under these conditions, the difference in iodine value of the products was 39 to 53 units, the difference increasing with the temperature until the flooding stage was approached. The best separation was obtained at a temperature of 135°.

A series of fractionations was made in which the yields of the extract fraction were 24, 36, 48, 68, and 80 percent. For the series as a whole, the difference in iodine values of the raffinate and extract fractions increased somewhat as the yield of the latter increased. This difference was approximately 48 units when the extract fraction amounted to 24 percent, and it increased to 53 units at 80 percent. The iodine value of the drying fraction ranged from 169 to 144, while that for the raffinate oil varied from 121 to 91.

An effort has been made recently to operate at lower solvent ratios and obtain more economical operation. The effect of mass velocity upon the fractionation of soybean oil was studied for three different feed rates. Ten parts of solvent to one of oil was used for all of the tests. Oil rates of 62, 92, and 122 pounds per hour per square foot of free cross-sectional area were used. It was necessary to operate at lower temperatures, but the degree of fractionation, as measured by the differences in iodine values of the products, changed surprisingly little. They were 38.1, 37.1, and 36.5, respectively for the oil rates given above. The reflux rate was adjusted to give exact yields of approximately 45 percent in each case.

RESEARCH AND MARKETING PROJECTS

Under the Research and Marketing Act of 1946 funds have been allotted recently for conducting work at the Northern Regional Research Laboratory under the following RMA Work Projects:

No. 127: Developing Industrial and Other Outlets for Grain (Alcohol, Fuels, Vitamins, etc.) and Evaluation of the Development of Motor Fuels in Experimental and Full-Scale Engines.

No. 128: Fundamental Studies on the Flavor Stability of Soybean Oil.

No. 145: Production of Antibiotics from Agricultural Sources.

No. 188: Development of Feed and Other Uses for Byproducts from Grain and Other Crop Adjuncts to Alcohol Production.

Line projects have been prepared for these work projects. The line projects do not include items already covered in our regular ARA projects.

Recruitment of the necessary personnel has been underway for some time and it is hoped that the staff will be rounded out in the near future. In the meantime, however, key men for each project have been transferred from the regular Northern Laboratory staff to form a nucleus for building up an RMA staff, and to initiate the work as rapidly as possible, to carry out the research investigations authorized by this Act.

AGRICULTURAL CHEMICAL RESEARCH DIVISION

L. F. Martin, Head

Administrative headquarters and three research sections of the Division are housed in the Southern Laboratory building, but approximately three-fourths of the experimental work is conducted at nine field stations. There is research in progress at New Orleans on some phases of all of the projects except those on tung nuts. Field stations engaged in work on citrus fruits are located at Winter Haven, Florida, and Weslaco, Texas, and on brining of cucumber and other vegetable products at Raleigh, North Carolina; work on sugarcane and sorgho is conducted at Baton Rouge and Houma, Louisiana, and at Auburn, Alabama, and seasonally at Meridian, Mississippi; work on sugar beets is carried out during the processing season in the beet producing areas and at other times at New Orleans; and tung nut investigations are in progress at Bogalusa, Louisiana, and Gainesville, Florida.

SOUTHERN FRUITS AND FRUIT BYPRODUCTS INVESTIGATIONS

Citrus Fruit Constituents

A phase of this work which has been completed for the current season is the systematic investigation of a large number of chemical substances observed by staining techniques at different stages during the ripening of citrus fruits. Suitable staining or color reaction tests were developed for twelve different classes of constituents. These were applied at monthly intervals from the time that small fruit had first set and attained a diameter of about 2 cm. until fully mature fruit was available. The tests were applied to oranges from a selected tree at Winter Haven, Florida, and to grapefruit from a tree at Weslaco, Texas. They show marked changes as the fruit grows and matures and a very conspicuous change when it is finally ripe. An example of such a test is that for aldehyde. In the smallest fruit the aldehyde appears to be concentrated near the tips of the vesicles, but during development it is found gradually spreading through more of the vesicle tissue. When the fruit attains ripeness aldehyde is found distributed throughout the tissues of the vesicles. The practical significance of this work is that three or four of these tests can be followed through next season and their value established as a means of determining when the fruit is approaching suitable maturity and particularly when it is at the best stage of maturity for canning.

The isolation and study of individual constituents have continued with the separation of numerous fractions of the extracts of non-volatile lipid material. Large quantities of pulp

and juice were prepared at Winter Haven, Florida, and Weslaco, Texas, and the total lipids adsorbed on celite. From these filter cakes the concentrated lipids were recovered by extraction with fat solvents. Other solvents such as methyl alcohol and acetone have been used to separate three compounds in pure crystalline form: a sterol, a sterol glycoside, and a flavanone. Compounds of these general classes are known to exist in citrus, but these particular compounds have not been described previously. Detailed study of their properties and structure is under way by analytical and synthetic procedures. When their chemical structure is definitely established it will be possible to investigate their chemical behavior under juice processing conditions, and to determine any effect they may have on quality of the products. Phosphatides, pigments, and saponifiable fats have also been separated, but less well characterized than the three crystalline compounds. Significant differences in composition between oranges and grapefruit have been found.

Microbiological Studies

Separation of organisms from cultures collected in citrus canning plant surveys has been effected. The bacteria have been identified and results of this part of the survey completed. Few bacteria survived, and none multiplied in the acid medium provided by citrus juice. Identification of yeasts and molds found in these cultures is nearing completion and results will be published soon.

Feed Yeast Production

Intensive development and research have continued on the process for producing torula feed yeast utilizing citrus feed press liquor as a culture medium. Additional continuous runs have been made in the large pilot plant at Orlando, Florida, with notable improvement in operating efficiency and yields. Toward the close of operation for the 1946-47 season it was found that yeast yields of 60 percent of the weight of carbohydrate could be attained by diluting the press liquor to a carbohydrate content of 2-3 percent. This lead has been followed, and other details are being worked out, in a laboratory scale fermentation system at Winter Haven, Florida. The plant at Orlando, Florida, was operated routinely by the canning company beyond the period of experimental operation by the Bureau staff. Several thousand pounds of yeast were produced, and about a thousand pounds have been furnished for feeding experiments being conducted by the Florida and Washington State Experiment Stations. Large scale pilot plant experiments are again scheduled for the latter part of the current citrus processing season.

SOUTHERN VEGETABLES AND VEGETABLE BYPRODUCTS INVESTIGATIONS

Microbiological Studies on Cucumber Pickles

Work at the Raleigh Station during the past year has been

concentrated on the identification of a large number of yeasts and obligate halophiles isolated from cultures taken from vats in which vigorous gaseous fermentation was occurring. This systematic mycological study is near completion and results are to be published as soon as possible. The relation of specific organisms of this type to formation of bloaters in cucumber pickle salt stock is to be investigated, employing pure cultures of organisms in controlled experiments.

BEET AND CANE SUGARS AND BYPRODUCTS CHEMICAL AND TECHNOLOGICAL INVESTIGATIONS

Aconitic Acid

The second year of commercial recovery of aconitate from cane molasses and production of aconitic acid as a valuable by-product brought substantial progress and expansion. The economic and technological success of this process has now been fully established. While no new mills were able to install equipment and initiate the process during the past grinding, the Raceland factory more than doubled its output. During grinding a total of 360,000 lbs. of calcium-magnesium aconitate was recovered and sold. Additional centrifugal equipment made it possible to collect the precipitated salt efficiently, and the over-all yield was 2 lbs. per ton of cane processed. An average rise of about 1 point in purity was effected in the processed "B" molasses. A small part of the total "B" molasses available was not processed for aconitate, and results were very gratifying, in view of the short production of molasses from the crop which was lower than usual in tonnage and Brix of the juice. Operation is to be extended, and an additional 75,000 to possibly 90,000 lbs. of aconitate will be obtained by processing final molasses acquired for this purpose. This can be done after the close of the grinding season; reconcentration of the processed molasses will be necessary, but this should prove profitable. It is hoped in this way to make available nearly half a million pounds of salt, from which about 200,000 lbs. of aconitic acid should be obtained to supply industrial demand. Aconitic acid is not currently available from any other source.

Sugarcane Wax

There is widespread and increasing interest in the possibilities of improved grades of sugarcane wax as a valuable by-product of the cane sugar industry. Much interest has been aroused by the distribution by the Sugar Research Foundation of the complete treatise on the subject prepared at the Houma Laboratory. Scores of inquiries have been received and numerous visitors interviewed from foreign countries, as well as from many parts of the

United States. At the present time American interests, other than those who have been operating Cuban extraction plants for the past four years, are considering initial operation on a pilot plant scale. Details of design and assembly of required pilot plant equipment, and technical advice and assistance have been provided by personnel of the Houma Laboratory.

Sugar Beet Storage

Generally encouraging results from large-scale tests of storage pile cooling last year have been followed by intensive investigation and engineering development. Steps have been taken by some beet sugar companies to put the method into commercial practice. This Division has cooperated with the Bureau of Plant Industry, Soils, and Agricultural Engineering, principally on accurate determinations of optimum temperatures to which beets should be cooled, and of the exact saving possible by cooling. The best arrangement for effectively and economically ventilating piled beets with cold night air is yet to be determined, but the value of the practice is well established.

Chemical and Microbiological Evaluation of Beet Sugars

The annual examination of campaign composite samples from all the beet sugar mills was carried out and the qualities factors, both chemical and microbiological, were issued in the usual form of a mimeographed publication. This report is distributed throughout the beet sugar industry, and to a number of other correspondents annually.

Research on Candy

During the year soybean processors greatly improved the quality of commercially available refined proteins. These products, consisting of 90 percent or more of soy protein, have recently been incorporated with fair success in hard candies. While clear hard candy cannot be produced, pulled candy pieces with a wrapper around the protein containing center have excellent flavor and appearance, and fair texture. Up to 7 percent protein can be so incorporated and provides adequate balance in candy that would otherwise be solely carbohydrate. The improved proteins are also more readily used in enriching creams, nougats, caramels and other types. Price of these new refined soy proteins will be a factor in their adoption by commercial candy manufacturers. Work has been done throughout the year on the testing of a wide variety of oils in a search for a possible substitute for mineral oil slab dressing. Cooperation is continuing with the National Confectioners' Association.

TUNG NUTS INVESTIGATIONS

Rapid Methods for Analysis of Tung Fruit

Especial attention has been given during the year to more

rapid methods of sampling and analyzing whole tung fruit. This work has been of importance in providing a basis for Commodity Credit purchasing of tung. Improved procedures were devised and their accuracy established by referee work. Personnel of the Tung Oil Laboratories are engaged in this work as members of a sub-committee of the American Oil Chemists' Society committee on analysis of oil seeds.

LABORATORY OF FRUIT AND VEGETABLE CHEMISTRY
LOS ANGELES, CALIFORNIA
E. A. Beavens, In Charge

CITRUS

Standardization of Desert Grapefruit Juice

Work on this project is now being concluded and the results of three seasons' experimentation and organoleptic tests are being compiled for publication. The following conclusions may be drawn from the results of this work:

1. Of the carbonate salts tested as the neutralizing agent, sodium bicarbonate was found to be the most satisfactory from the standpoint of cost and acceptance by Food and Drug officials.
2. Sucrose was found to be better than dextrose (corn sugar) for the sweetening agent.
3. The best juices were those standardized to an acidity of pH 3.2-3.4, and a solids content of 12.5°-13.0° Brix.
4. Organoleptic tests on two seasons' samples prepared each month from October to July have shown a definite preference for the standardized juices.
5. The addition of the bicarbonate salt did not cause any loss of vitamin C.

Freezing Preservation of Orange Puree

Samples of California and Arizona Valencia oranges and California Navel oranges have been processed through a puree machine, mixed with cane sugar at the ratio of 5 parts fruit to 1 part sugar, and quick-frozen at -25° F. After nine months storage at 0° F., these frozen purees were made up into test lots of orange sherbet by one of the local ice cream companies. All three sherbets were judged to be of excellent quality with no evidence of a strong "terpeney" flavor. Even the Navel orange sherbet was good with no bitter flavor which is generally produced when this variety of orange is used. These preliminary experiments were so successful that a local freezing company specializing in fruit purees packed a large quantity of frozen orange puree last season for use in making orange sherbet. Shipments were made to two large ice cream companies in the East, and two Los Angeles companies, such as the Ralph's chain stores, are now making orange sherbet from the frozen puree. Frozen orange puree is a new contribution to the field of utilization of cull citrus fruits.

Keeping Quality of Chilled, Fresh Orange Juice

During the past orange season, the processing and distribution of fresh, refrigerated orange juice has become firmly

established in Southern California. Laboratory experiments have conclusively shown that the storage life of this type juice can be materially increased if the juice is first deaerated, chilled to 30° F. as quickly as possible, packaged in airtight containers without leaving any headspace, and stored at a temperature of 27-30° F. Under these conditions, unpasteurized juices could be kept as long as 14 days with little loss of fresh flavor, and no loss of vitamin C. Juices kept at these temperatures as long as 21 days were judged to be superior in flavor to most commercially canned juices now on the market.

After comparing the flavor of deaerated and undeaerated fresh orange juice stored at 30° F. for 12-14 days, one of the largest packers of this type of juice immediately installed deaeration equipment. Another large packer now cools his juice to 30° F. and ships it as far as New York City at this temperature, with no loss of any shipments and no complaints of stale flavor from the consumers.

Frozen Grapefruit and Orange Segments

Work has been continued on the freezing preservation of grapefruit segments to determine what months during the growing season are the most desirable to pack this type of product. Frozen segments prepared from grapefruit picked before the latter part of January were poor in quality since they were bitter and lacked a characteristic fruity flavor.

Lots of mixed grapefruit and Navel orange segments packed in sweetened and unsweetened grapefruit juice and in sugar syrups were sampled after 9 and 12 month storage periods at 0° F. These mixed segments were found to be of excellent flavor, color and texture, with no bitterness apparent in the Navel sections. This product has an excellent eye appeal because of the brilliant orange colored segments. Navel orange segments packed in Navel orange juice were distinctly bitter in flavor. Apparently the bitter substances in the Navel orange are located in the peel and get into the juice as a result of the reaming process. By removing the segments from the peel and section membranes and freezing in grapefruit juice or water syrups, no bitterness develops.

Heat Processing of Grapefruit and Navel Orange Segments

California-Arizona Desert grapefruit segments have never been commercially heat processed in quantities since they are very tender in texture and are easily broken up as a result of the heat treatment. If heat processed segments could be successfully packed and distributed without excessive damage to their texture, it would open a new outlet for the utilization of grapefruit and possibly Navel oranges. Samples of grapefruit segments were packed in hermetically sealed cans and heat processed in hot water at 200° F. for 2-1/2 minutes. These samples were then examined for firmness with varying degrees of success, depending

on the maturity of the fruit and other uncontrolled factors. Since canned tomatoes have been successfully firmed by the addition of small amounts of calcium salts, it was thought this treatment might work on the grapefruit segments. Samples were processed with added calcium salts and similar quantities of the salt plus low-methoxyl pectin. On the basis of drained weights and tasting, it was evident that the added calcium had definitely aided in firming the segments. The best results were obtained with the calcium salt-pectin mixture which reduced the drained weight losses by 50 percent.

Mixed Navel orange and grapefruit segments were packaged in sealed containers and heat processed in hot water at 200° F. without the addition of any firming agents. After eight months storage at room temperature, these segments were still well flavored with no evidence of bitterness from the Navel orange. Other packs of straight Navel segments put up in sugar syrups and sweetened Navel orange juice were similarly heat processed and sampled after eight months storage. The segments in the water syrups were well flavored while those put up in the Navel juices were extremely bitter and not fit to eat.

VEGETABLE PRODUCTS

Improvements in the Quality of Canned Tomato Paste

This work was undertaken to study the chemical composition and physical characteristics of commercially canned tomato paste as a basis for judging quality in samples prepared under present day methods of processing. Samples of tomato paste are being prepared in the pilot plant under controlled conditions to determine what improvements in quality can be made. The development of more suitable and accurate methods of analyzing tomato paste as a measure of quality is being undertaken. Considerable time has been spent in working out a method for determining the iron and copper contents of the samples, both spectrographically and colorimetrically. Chromatographic methods for determining the lycopene and carotene content in tomato paste have been developed as well as methods for evaluating the color of the samples. A method for measuring consistency of tomato paste has never been available to the industry. A "penetrometer" fitted with a newly designed penetration cone has been adapted to make these measurements, and its use has proven to be highly satisfactory.

RESEARCH AND MARKETING ACT OF 1946

An appropriation of \$250,000 has been made from R&MA funds for the construction and equipment of a laboratory at Pasadena, California, near the California Institute of Technology. This new laboratory will provide quarters for the Bureau's Laboratory of Fruit and Vegetable Chemistry which has been housed in rented space in Los Angeles since 1914. The new laboratory will be known as "Research Laboratory of The Bureau of Agricultural and Industrial Chemistry." Plans have been drawn, and bids for its construction were opened on February 5, 1948. It is anticipated that the new laboratory will be ready for occupancy within a year.

R&MA financial work on "New and Improved Uses of Citrus Products" will be a part of the program at the new laboratory.

FRUIT AND VEGETABLE PRODUCTS LABORATORY
PULLMAN, WASHINGTON
A. M. Neubert, In Charge

FRUIT PRODUCTS

Freestone Peach Canning and Freezing Studies

Studies on the relation between harvest maturity and quality of canned and frozen freestone peaches showed that there is no advantage, insofar as the quality of these products is concerned, in permitting either the Elberta or J. H. Hale varieties to become soft-ripe on the tree. A slightly lower quality resulted when these varieties were harvested at maturities requiring more than 8 days to ripen, and there was considerable loss in quality when 11 or more days were required for the fruit to ripen. Losses from bruising caused reduced yields of product when the fruit was permitted to soften on the tree. Decreased yields also resulted when the fruit was harvested on the immature side, because such fruit had not reached full size at harvest, and also because of wilt during ripening.

Results of a study on the effect of ripening procedure on the quality of the canned product showed that fruit ripened at about 75 deg. F., under conditions of either moderate or high humidity, developed a better flavor and retained good flavor over a longer period than that ripened at higher temperatures. Preparation of a manuscript covering the technical aspects of the freestone peach processing industry in Washington was undertaken in cooperation with the Irrigation Branch Station and Department of Horticulture, State College of Washington, and the Western Regional Research Laboratory.

An investigation in size-grading freestone peaches showed mechanical grading to be commercially practical provided certain precautions were taken in handling the fruit, and suitable sizing equipment was used. To avoid excessive bruising, it was found necessary to size and sort peaches within 24 hours of harvest. Greater delay resulted in progressively increasing losses from bruising. Diverging belt-type graders were found incapable of sizing fruit with sufficient accuracy to permit packing one size of halves from a given size lot. A modified weight-type grader, capable of accurate size grading, was limited in capacity, and large, multiple units would be necessary for commercial operations. The use of mechanical size grading would permit greatly increased efficiency in the operation of freestone peach processing plants. These studies were conducted under a cooperative agreement with the BPISAE Laboratory, Wenatchee, Washington; Wenatchee Foods, Inc., Wenatchee, Washington; Storr Food Products Company, Yakima, Washington; and Food Machinery Corporation, Wenatchee Branch.

Canning Investigations on Apricots

Studies on the suitability for canning preservation of apricot varieties grown in Central Washington were continued in cooperation with the Irrigation Branch Station, State College of Washington. Studies on the relation of maturity at harvest and method of ripening on canning quality were also continued. Results of these studies will not be available until quality evaluations can be completed and the data summarized.

Detartration Studies on Grape Juice

Studies on detartration of Concord grape juice and the use of ion-exchange resins in controlling acidity were continued. Ion-exchange treatment was found to slightly increase the ratios, P_2O_5 - Mg O and P_2O_5 - Ca O, but the main effect was the removal of potassium ions. The only differences noted in juice quality as a result of ion-exchange treatment was an increase in acidity and a shift in color from blue to red which accompanied this increased acidity. Studies were continued on the problem of resin degeneration during use. A solution of the resin degeneration problem would permit use of this process by Pacific Northwest grape juice packers for accurately controlling the acid content of this product, and would also permit substitution of bulk-tank refrigerated juice storage in place of the present method of storage in 5-gallon carboys.

VEGETABLE PRODUCTS

Froth Flotation Process

Considerable time was devoted to consultation with pea processors on the application of the froth flotation process to the cleaning of vined green peas. Fifteen froth flotation units were constructed and operated by pea processors in the eastern Washington and Oregon pea areas. These units, costing from \$2500 to \$5000 each, and representing a combined capacity for cleaning 34 tons of peas per hour, were used in both canning and freezing plants. Value of the process to the industry in 1947 is estimated at over \$300,000, with a probable annual value of about \$2,000,000, when the process becomes fully exploited by the entire industry.

Canning Technology of Pacific Northwest Vegetables

Studies on the canning quality of vegetables grown in the Lewiston area of Idaho were continued in cooperation with the Department of Horticulture, University of Idaho. Special attention was given to the effect of fertilizer treatment on the canning quality of tomatoes. Results of these studies will not be available until quality evaluations can be completed and the data summarized.

Freezing Technology of Pacific Northwest Vegetables

Studies on the freezing preservation of sweet corn were continued in cooperation with the Department of Horticulture, University of Idaho and the Western Regional Research Laboratory. Emphasis was placed on the relation of planting date and irrigation program on maturing characteristics of the Golden Cross Bantam variety. Information on the yield and processing quality of twelve varieties of corn, as grown in the Lewiston, Idaho, district, was released to the industry.

ENZYME RESEARCH LABORATORY
Arnold Kent Balls, Head Chemist

This Laboratory is engaged in basic research on the natural catalytic substances in living tissue which determine its growth, ripening and decay, and on the differences the natural catalysts make in the size, ripeness and shipping qualities of agricultural products. Full maturity and ripeness are generally desirable in any agricultural product, but quite often they also predispose the material to decay and thus interfere with shipping and keeping qualities. The present methods of handling foods are frequently a compromise between ripeness and resistance to decay in storage or transit.

Much of the work of the Enzyme Research Laboratory deals with the basic chemistry of ripening, which is in turn largely concerned with the changes that starches, sugars and in some cases also fats undergo in the ripening agricultural material.

Artificial Stimulation of Ripening: Experiments on the artificial stimulation of ripening in citrus fruits have been conducted for several years. The California Experiment Station has helped us tremendously in this work. Such artificial ripening decreases the bitter taste acquired by Navel orange juice on standing, but so far it has apparently produced other off flavors instead.

Artificial stimulation of ripening in freshly harvested rice appears to decrease somewhat the heating of the grain in the storage bins, and is being tried out commercially in a small way. A similar effect was found with prematurely harvested wheat. There is some evidence here of an immediate "aging" of the wheat, permitting it to be milled sooner than would normally be possible.

The Bitter Principle of Navel Oranges: In line with the foregoing research, the bitter substance (limonin) that forms in Navel orange juice is being investigated chemically. Its structural formula, so far as we have worked it out, has offered a clue to the way this substance is formed and what its precursor in the juice may be.

Enzymes Causing Changes in Starches, Sugars and Fats: All these enzymes play a part in the process of ripening, and many of them are being studied. The enzymes in grains that change starch to sugar are receiving considerable attention, and a similar starch-digesting enzyme from sweet potatoes has been crystallized, so its action in the pure state can now be studied. The alpha-amylase of malt has also been prepared in a much purer form than ever before attained although the protein has not yet crystallized out.

The build-up of sugar in citrus fruits evidently involves the action of phosphates in the plant, and a new reaction whereby phosphate is transferred in oranges from one substance to another has been discovered and is being studied. This new biological reaction seems to be a rather general one throughout both plant and animal nature.

The role of fats in agricultural chemistry has also been considered in a study of the substances known as phospholipids. While these are present throughout nature, it is very difficult and often impossible to separate them from one another. To overcome this difficulty, one such substance (dipalmitoyl cephalin) has been synthesized in the laboratory and is now being studied. The preparation of this compound, now available for the first time, was a difficult synthesis of which we are quite proud.

Effect of High Pressures on the Enzymes of Agricultural Products: From theoretical considerations it appears probable that extremely high pressures may be expected to activate and/or inactivate enzymes. Different enzymes should, however, be affected at different pressure levels, thus offering an opportunity to separate them, and perhaps to get rid of undesirable enzymes in many biological products such as sera and similar medical preparations.

In order to investigate this idea a rather complicated apparatus has been built that can subject about 25 cc. of a liquid to pressures as high as 130,000 pounds per square inch for long periods of time. The machine has just recently been completed. Preliminary experiments indicate that profound effects are in fact produced on enzymes by such enormous pressures, but we have no details to report yet.

DIVISION OF BIOLOGICALLY ACTIVE COMPOUNDS

Thomas D. Fontaine, Acting Head

BASIC INVESTIGATIONS IN THE CHEMISTRY OF AGRICULTURAL PRODUCTS

Investigations on the Biochemistry of Plant Disease Resistance

It has been established that tomato plant juice contains an antibiotic(s) which inhibits the growth of the specific tomato wilt causing organism, Fusarium oxysporum f. lycopersici. The antibiotic activity of crude tomato plant extracts has been designated as "tomatin", whereas one crystalline compound isolated, possessing the antifungal properties of crude tomatin concentrates but showing very low antibacterial activity, has been designated as "tomatine". Crystalline tomatine has been tentatively identified as a glycosidal alkaloid, consisting of an aglycone portion (designated as "tomatidine") and a carbohydrate fraction. Tomatine and tomatidine have been shown to be different from solanine and solanidine (the latter two obtained from the Irish potato plant). The antibiotic activity of tomatine has been found to reside in the aglycone portion (tomatidine) of its molecule.

Since crystalline tomatine and tomatidine showed considerable antibiotic activity toward certain of the fungi which cause disease in man, they are of interest from a medicinal standpoint. The pharmacological and clinical studies on these compounds are in progress.

Rutin has been isolated from tomato plant extracts during experiments designed to purify the antibiotic factors present in such extracts. The isolation of rutin was accomplished by chromatographic procedures and because rutin and the antibiotic activity of crude tomatin concentrate were eluted from the column together an early indication of the nature of tomatine was indicated. In certain instances it was found that tomato plant juices failed to inhibit certain organisms which were readily inhibited by crystalline tomatine. The testing of tomatine in the presence of rutin or quercetin demonstrated that they had an antagonistic effect on the antibiotic activity of tomatine. It has been recognized that tomato plant extracts contain stimulators as well as inhibitors and the importance of these and other factors are being investigated in relation to plant disease resistance.

Investigations on the Mechanism of Action of Plant-Growth Regulating Substances

A growth-regulating compound, 2-iodo-3-nitrobenzoic acid, containing radioactive iodine has been used in these investigations. The compound has been designated as "INBA". By using radioactive INBA as a tracer it has been possible to demonstrate that the plant-growth regulator enters the plant and accumulates in the actively

growing tissues; is probably associated with the carbohydrate fraction; and can be recovered from the plant as the intact molecule. It has been shown also that a non-essential metabolite, such as INBA, is concentrated in the tissue of an artificially induced tumor in the bean seedling when the growth regulator is applied to a leaf. The differences in the action of growth regulators toward dicotyledonous and monocotyledonous plants are being investigated.

Potential plant-growth regulators have been synthesized and treated for activity. A new plant-growth regulator, having approximately 95 percent of the plant-growth regulating activity of 2,4-dichlorophenoxyacetic acid (the well known weed killer), has been synthesized. The new compound can be made to contain radioactive iodine and it will now be possible to investigate the mode of action of a typical halogenated phenoxyacetic acid.

PHARMACOLOGY DIVISION, ALBANY, CALIFORNIA
Floyd DeEds, Head

PHARMACOLOGY INVESTIGATIONS

Azopenicillin.

A sample of azopenicillin was submitted by the Northern Regional Research Laboratory for determination of toxicity. Due to difficulties in preparing a suitable solution of azopenicillin for intravenous administration only a limited study has been made. Using an aqueous solution of azopenicillin, and working as rapidly as possible to avoid difficulties due to jelling, a tentative determination of the acute toxicity has been made in 22 white mice. The dose required to kill 50 per cent (LD_{50}) of the mice was found to be about 126 mgm. per kilogram of body weight. Welch and co-workers have reported the LD_{50} of sodium penicillin to be about 1600 mgm. per kilogram of body weight in mice. Assuming the two strains of mice to be comparable, and assuming that slight jelling of the solution played no role azopenicillin appears to be about 12 times more toxic than sodium penicillin. However, these results are only preliminary and it may be that a more suitable solvent will give different results.

Nicotine Derivatives.

1-nicotine and five derivatives have been compared with regard to their ability to produce the same rise in blood pressure of dogs. These observations were made in dogs incidental to other experimental procedures and are therefore of a preliminary nature. All the compounds were dissolved in water with the aid of hydrochloric acid and the pH adjusted to approximately 7 with the aid of a glass electrode. The compounds were administered intravenously to dogs under barbital anesthesia, and all doses were calculated in terms of mgm. per kilogram of body weight. If a value of 1 is assigned to 1-nicotine, the relative quantities of the six compounds required to produce the same rise in blood pressure are as follows: 1-nicotine--1, metanicotine--5, d-1-nornicotine--6, n-methyl myosmine--40, nicotyrine--160, myosmine--500.

Using oral administration in rats the acute toxicity of nicotyrine was determined. A dose of 1175 mgm. per kilogram of body weight killed 50 per cent of the animals. Oral administration to rats of 1000 mgm. per kilogram of body weight produced severe chromodacryorrhea (bloody tears).

Lauryl Gallate.

The chronic toxicity of lauryl gallate has been studied by feeding groups of rats a control diet and diets containing 0.006, 0.012, 0.025, 0.05, 0.1, 0.25, 0.5, 1.0, 2.5, and 5.0 per cent of the compound. Feeding of these diets for 100 days produced no effects on the growth rate or general appearance of the animals when the concentration of lauryl gallate was 0.5 per cent or less. Growth rate was retarded by 1 per cent and all animals receiving 2.5 or 5 per cent died within 10 days.

Rutin.

Investigations on the mechanism of action of rutin have con-

tinued. Studies on the effect of rutin on the oxidation of epinephrine, by the Warburg technic, have shown that rutin is far more effective in preventing the autooxidation than in retarding oxidation catalyzed by Cu^{++} or amine oxidase.

Preliminary experiments have shown that rutin and its aglycone quercetin have a sparing action on suboptimal amounts of ascorbic acid when administered to scorbutic guinea pigs.

Trypan blue injected intravenously in rabbits escapes from skin capillaries slowly under normal conditions but escapes rapidly in areas of irritation induced experimentally. Following injection of rutin the escape of dye in irritated areas is definitely retarded, thus demonstrating a direct or indirect effect on the capillaries.

Cottonseed Meal and Pigment Glands.

Hexane extracted meal has been shown to be more toxic than defatted-depigmented cottonseed meal. Feeding experiments on albino rats have shown that 0.25 per cent pigment glands added to the basic diet interferes with reproduction. Histological studies demonstrated that this decreased fertility is due in part if not wholly to depressed spermatogenesis.

Subtilin.

Investigations on the antibiotic subtilin have shown that red blood cells are agglutinated in vitro. Following subcutaneous or intramuscular injection absorption is too slow to permit attainment of therapeutic concentrations in the blood stream. Slow intravenous infusion of subtilin will yield desirable blood levels, but the rate of infusion must be slow enough to avoid precipitation of subtilin and the production of anaphylactoid reactions.

MICROBIOLOGY RESEARCH DIVISION
Located at
Eastern Regional Research Laboratory

Mathilde Solowey, Acting Head of Division

MICROBIOLOGY OF DRIED EGGS AND RELATED EGG PRODUCTS *

Heat Resistance of *Salmonella* Organisms Isolated from Spray-Dried Whole-Egg Powder

The Decimal Reduction Time was applied to determine the relative heat resistance of 95 strains (24 types) of *Salmonella* and other Gram-negative organisms suspended in liquid whole egg and in tryptose broth. With the exception of 4 strains, the DRT (90% kill) for all the organisms was less than one minute at 58° C. The data add further support to the practical application of pasteurization of liquid egg for reduction in the numbers of undesirable organisms.

Mold in Spray-Dried Whole-Egg Powder

Examination of more than 6,000 samples of high-moisture (4 to 6%) spray-dried whole-egg powder for presence of molds revealed that 36% of the samples had mold counts of 100 or higher per gram; only 1.3%, however, had counts of 1,000 or higher. Incidence of molds in the samples varied with the season of year as well as with individual dehydration plants. *Aspergilli* were most frequently isolated from high mold count samples (1,000 per gram or higher). Comparative growth of the different molds as a function of the incubation temperature was also investigated.

Aerobic Mesophilic Sporeforming Bacilli Isolated from Egg Powder

Taxonomic studies were made on 279 cultures isolated from 177 samples of egg powder representing lots produced in 32 dehydration plants. The cultures were picked from tryptone-glucose-extract agar and dextrose-tryptone agar plates incubated at 55° C. Two hundred and seventy-two of the cultures were identified as *B. subtilis*, the remainder as *B. megatherium* or *B. brevis*. It is indicated that the presence of aerobic mesophilic sporeforming bacteria in egg powder which are culturable at 55° C. may reflect on the quality of the shell egg dried and their presence may hasten spoilage of food products and food dishes in which the contaminated powder is incorporated.

* These investigations were undertaken in cooperation with the Poultry Branch, Production and Marketing Administration, U. S. Department of Agriculture, Washington, D. C. and/or the Quartermaster Food and Container Institute for the Armed Forces, Chicago, Illinois.

An Investigation of Hypochlorite Solutions as Used in the Egg-Breaking and Dehydration Industries

Investigations on the germicidal values of disinfectants used by food industries in the sterilization of processing rooms, equipment and utensils have to date proceeded along the following lines: (1) the influence of liquid whole egg upon the available chlorine in solutions of sodium hypochlorite, and (2) the influence of liquid whole egg on the germicidal efficiency of hypochlorite solutions. Results indicate that chlorine residuals are not good indicators of bactericidal activity for solutions of hypochlorites containing appreciable amounts of liquid whole egg. Concentrations of liquid whole egg and exposure time are two of the more important factors which influence bactericidal efficiency of a hypochlorite, other factors being pH, temperature and agitation of the solution.

RMA Projects

One RMA project has been approved for this Division to date, i. e., "Quality Preservation of Poultry Products, Improved Products, and Improved Techniques in Maintaining Quality in Storage" (Line Project No. RMA Part II - 112-MRD-1, "Improvement of Microbiological Methods for the Detection of Salmonella Organisms in Poultry Products"). In view of the fact that the funds for this work have only recently been made available, the activity so far has been concerned only with recruitment of personnel and purchase of equipment.

NAVAL STORES RESEARCH DIVISION
E. L. Patton, Head

Approximately two-thirds of the research of this Division is carried out at New Orleans; the remainder at the Naval Stores Station, Olustee, Florida.

INVESTIGATION OF NAVAL STORES PRODUCTION,
PROCESSES AND EQUIPMENT

Processing of Pine Gum

Research in processing has centered on improvement of the Division's process for the continuous flash distillation of pine gum. This process, first developed in 1945, has proved applicable to the distillation of undiluted, cleaned pine gum, as well as to the lighter diluted gum for which it was developed. Use of the continuous flash still to strip solvent at atmospheric pressure from any reasonably stable solution appears possible, and several inquiries have been received suggesting further investigation.

Construction has been started on a model of the continuous flash still of commercial size to demonstrate the lower production costs and improved quality of product obtained by this system. The new still should be ready for operation during the summer of 1948.

Studies of Cup and Gutter Materials

Investigation has continued to determine the materials most suitable for turpentine cups and gutters when used with the Forest Service system of sulfuric acid stimulation of pine trees. A suitable material must withstand not only the corrosive action of sulfuric acid, but also the considerable amount of mechanical abuse found in ordinary turpentine operations. The perfect material has not been found, but information has been gained on the useful life of commercial cup materials under acid stimulation. Vitreous enamel, nickel, monel, and various resin-coated cups have been further tested with different degrees of success. Additional testing is necessary before the proper cup material can be selected.

INVESTIGATION OF THE COMPOSITION, COMPONENTS,
AND DERIVATIVES OF NAVAL STORES

Synthetic Elastomers from Turpentine Derivatives

Products of gum turpentine, such as isoprene and myrcene derived from alpha- and beta-pinene, respectively, have been

copolymerized with styrene under a wide variety of conditions. High-quality synthetic elastomers have been produced in good yield utilizing isoprene from turpentine.

Chemical Derivatives of the Pinenes

Work has been continued on the development of useful chemical derivatives of alpha- and beta-pinene. These include hydration products and chlorinated and silicon-containing compounds. Basic information on the reactions and structures of these substances is being accumulated with the ultimate object of developing new industrial uses for turpentine.

Composition Studies

The composition of various commercial terpene products was determined by careful fractional distillation and examination of the physical properties of the fractions obtained. Methods have been developed for determining important impurities, namely, acids and peroxides, in turpentine.

INVESTIGATION OF THE USES OF NAVAL STORES

Purification of Dehydrogenated Rosin

Increasingly large quantities of catalytically dehydrogenated rosin are being used in the manufacture of synthetic rubber, specifically GR-S 10. Certain impurities present in such rosin decrease the rate of polymerization and/or the yield of polymer. But it was found that these undesirable impurities were removed if the dehydrogenated rosin was dissolved in petroleum naphtha and percolated through towers of fuller's earth or other adsorbent silicates. The improved rosin gives better yields of polymer when used as an emulsifier. (The impurities adsorbed on the fuller's earth are easily dissolved off with alcohol so that the adsorbent can be used over and over again).

The use of the alkyl esters of the maleic anhydride addition product of levopimaric acid as emulsifiers for synthetic rubber is now being investigated. Polymers having considerably higher elongations and tensile strengths have been prepared with these emulsifiers than with the commonly used emulsifiers.

Utilization of Resin Acids

Considerable work has been done on the preparation of the maleic anhydride adduct of levopimaric acid from pine gum. The conditions necessary for the nearly quantitative separation of this adduct have been determined. It has also been found that by esterifying the mixed mono- and diglycerides of linseed oil with the addition product,

varnishes having excellent hardness and water resistance can be prepared. Further studies are under way on the effect of acid stimulation of pine trees on the composition of the gum and the effect of such changes, if any, on the utilization of the rosin obtained.

Studies have also been carried out on the composition of the pine gum produced by fungus-stimulated trees.

The separation, purification, and identification of the acidic components of pine gum and rosin have been studied.

Preparation of Rosin Esters and Metallic Resinates

It is desirable to reduce the acidity of rosin which is to be used in varnishes and surface coatings. This is usually done by reacting the rosin with a polyhydric alcohol such as glycerol, or with a metallic oxide such as calcium or zinc oxide. The optimum conditions for carrying out such reactions have been investigated and the physical and chemical properties of the resins formed have been determined.

INVESTIGATIONS UNDER RESEARCH AND MARKETING ACT

RMA-100 Pilot Plant Production of Gum Naval Stores Derivatives

Research was begun in October 1947, on pilot plant production of gum naval stores derivatives to develop new products obtainable from pine gum, other than the traditional turpentine and rosin. The first product selected for pilot plant development was the maleic anhydride adduct of levopimaric acid, derived from undistilled pine gum. This adduct should prove useful in the preparation of high-quality surface coatings and other industrial uses requiring a high melting point bi-functional compound. Laboratory production of the adduct was developed by this Division in 1946. It is the intention of this project to determine production costs, necessary equipment, and otherwise to commercially evaluate new products which may be made easily by naval stores processors already in operation.

NATURAL RUBBER EXTRACTION & PROCESSING INVESTIGATIONS
U. S. NATURAL RUBBER RESEARCH STATION
SALINAS, CALIFORNIA

I. C. Fuestel, Head

The development of sources of rubber either within the United States or in areas in the Western Hemisphere where lines of supply can be protected by our armed forces, has been recognized as essential to our national security. The Stockpiling Act directed the Secretary of Agriculture to conduct research on agricultural materials declared strategic and critical under the provisions of that Act. Rubber was so designated, and appropriations were requested to conduct the necessary research.

The Emergency Rubber Project of the Department of Agriculture, which conducted research on the production of domestic rubber during the war, was liquidated in 1946. Subsequently, the Navy Department, through its Office of Naval Research, provided funds for an interim program of research, pending the time when the Department of Agriculture could obtain appropriations. A contract to conduct research on guayule was arranged by the Office of Naval Research with Stanford University. This contract was terminated on July 31, 1947.

The Department of Agriculture again took over the work on August 1, 1947, the Bureau of Agricultural and Industrial Chemistry being designated to conduct research on rubber extraction, processing, and byproducts; and the Bureau of Plant Industry, Soils, and Agricultural Engineering conducting research on all phases of crop production. Guayule and Kok-saghyz were regarded as outstanding in their promise for domestic rubber production. Of the two, guayule shows the greater immediate promise of high yield and low-cost production and will receive major attention. Research on guayule is based on the results of intensive wartime studies which point clearly toward the objectives of lower production costs, higher yields, and increased efficiency in recovering the available rubber. The work of this Bureau's Laboratory is divided into four sections: The Latex Section will develop methods for the recovery of natural rubber in latex form. The Laboratory Extractions Section will conduct chemical and technological research on the extraction of rubber, resins, and other constituents; research on methods of recovery of the rubber in coagulated form; retting solvent extractions; and separation of rubber hydrocarbons and resins. The Pilot Plant Operations Section will engage in developmental work on a pilot plant scale for the recovery of rubber; the perfection of various processing steps; and the development of new and improved methods for extraction operations. The Analytical and Physical Testing Section

will perform service and control chemical analyses and tests for the other sections, as well as research on the development of new or improved methods for the chemical analysis of constituents of rubber-bearing plants, and the physical testing of various rubber products. The latter section will also engage in a search for special uses for which the domestically produced rubber products may be utilized to best advantage.

Cooperative arrangements have been tentatively negotiated with the Bureau of Plant Industry, Soils and Agricultural Engineering, relating to planting, maintenance and harvesting of guayule shrub for experimental use of this laboratory, and to other matters of mutual concern.

Much time has been spent in reassembling and repairing equipment and collecting necessary supplies; also in recruiting competent personnel, particularly group leaders and other staff members previously engaged in rubber extraction and processing investigations under the Emergency Rubber Project. Approximately half of the proposed staff has now been appointed.

